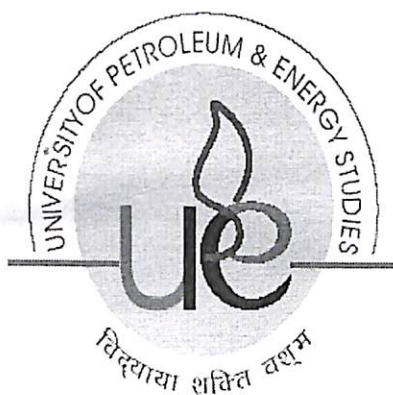


Scale Up of Hydrazine Hydrate Process from Bench Scale to Pilot Scale
at IICT, CSIR

By

Ibrahim Khan



College of Engineering
University of Petroleum & Energy Studies

Dehradun

April, 2011

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SCALE UP OF HYDRAZINE HYDRATE PROCESS
FROM BENCH SCALE TO PILOT SCALE

AT IICT, CSIR

A thesis submitted in partial fulfilment of the requirements for the Degree of
Master of Technology
(Process Design Engineering)

By

Ibrahim Khan

Under the guidance of

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CERTIFICATE

This is to certify that the work contained in this thesis titled “Scale Up Hydrazine Hydrate Process from bench scale to pilot scale” has to be carried out by **Ibrahim Khan** under our supervision and has not been submitted elsewhere for a degree.



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Abstract

A chemical company[†] is considering the production of Hydrazine Hydrate. The company has tied up with Indian Institute of Chemical Technology (IICT), Hyderabad, for development of Hydrazine Technology.

IICT, the premier chemical institute of India, will develop a bench scale process for manufacturing hydrazine hydrate and also demonstrate at pilot scale. It would provide detailed designs for a commercial plant with 8,000 tonne per annum capacity. Hydrazine hydrate is extensively used in the pharmaceutical, agrochemical and water treatment space.

The process, being developed by IICT, Hyderabad, is based on hydrogen peroxide route, which is considered environmentally benign.

The scope of the project is to develop a design and scale up to produce 12 kg/hr continuous on Pilot Scale of Hydrazine Hydrate using the new Hydrazine technology of IICT.

[†]*Subject to confidentiality*

Keywords: Hydrazine Hydrate; Pilot plant; Equipment Design; Heat exchanger design; Scale-up

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Everything happens only with the grace of God. I thank the Almighty God for providing me an opportunity, strength and ambience to work on this Project

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Ibrahim Khan

Table of Contents

CONTENTS	Page No.
ABSTRACT	i
LIST OF FIGURES	viii
LIST OF TABLES	x
NOMENCLATURE	xi
1 INTRODUCTION	1
1.1 System Considered	1
1.2 Scope and objective of the Project	1
2. LITERATURE REVIEW	2
2.1 Hydrazine: Timeline and Application	2
2.2 Properties.....	3
2.3 Methods of Production	6
2.4 Selection of Process	8
2.5 Selected Process Description	9
2.6 Process Synthesis	11
3. DESIGN DATA ESTIMATION	13
3.1 Properties Estimation of the Components	13
4. PROCESS DESIGN ASPECTS	17
4.1 MOC Philosophy	17

5. EQUIPMENT DESIGN	19
5.1 Storage Tanks	19
5.2 Stirred Vessels	21
5.3 Distillation Columns-Packed Tower	25
5.4 Crystallizers Sizing	33
6. PROCESS DESIGN OF PIPING SYSTEM	37
6.1 Line Sizing	37
6.2 Orifice Sizing	42
6.3 Pumps Selection	44
7. HEAT EXCHAGER DESIGN	45
7.1 Horizontal Condenser -Thermal Design	45
7.2 Thermosyphon Reboiler -Thermal Design	53
8. SCALE UP	62
9. CONCLUSIONS	69
10. RECOMMENDATIONS	69
BIBLIOGRAPHY	70
APPENDIX 1: About IICT	72
APPENDIX 2: Spreadsheets	73
1. Line Sizing	
2. Packed Column Sizing	
3. Horizontal Condenser	
4. Orifice Sizing	
5. Thermosyphon reboiler	
6. Tank Sizing as per IS-4179	
APPENDIX 4: Property Estimation	79
APPENDIX 3: Project Timeline	92

LIST OF FIGURES

Figure No.	Page No.
2.1 Basic flow diagram	9
2.2 Process flow diagram	9
3.25 Densities of MEK-Azine and HH Vs Temperature	15
3.26 Viscosities of MEK-Azine and HH Vs Temperature	15
5.1 T-xy Diagram for T-304	26
5.2 Input to Mapple Code for No. of Equilibrium Stages	28
5.3 Mc-Cabe Thiele Digram for T-304	39
6.1 Liquid Orifice Sizing	43
6.2 Vapor Orifice Sizing	43
7.1 Inputs for Spreadsheet for Design of Horizontal Condenser	51
7.2 Result from Spreadsheet for Design of Horizontal Condenser	52
7.3 Pressure drop Vs Recirculation ratio	57
3.1 Density of H ₂ O ₂ Vs Temperature	79
3.2 Viscosity of H ₂ O ₂ Vs Temperature	79
3.3 Specific Heat of H ₂ O ₂ Vs Temperature	80
3.4 Thermal Conductivity of H ₂ O ₂ Vs Temperature	80
3.5 Density of MEK Vs Temperature	81
3.6 Viscosity of MEK Vs Temperature	81
3.7 Specific Heat of MEK Vs Temperature	82
3.8 Thermal Conductivity of MEK Vs Temperature	82

3.9 Density of Acetamide Vs Temperature	83
3.10 Viscosity of Acetamide Vs Temperature	83
3.11 Specific Heat of Acetamide Vs Temperature	84
3.12 Thermal Conductivity of Acetamide Vs Temperature	84
3.13 Density of Water Vs Temperature	85
3.14 Viscosity of Water Vs Temperature	85
3.15 Specific Heat of Water Vs Temperature	86
3.16 Thermal Conductivity of Water Vs Temperature	86
3.17 Density of EDTA Vs Temperature	87
3.18 Viscosity of EDTA Vs Temperature	87
3.19 Specific Heat of EDTA Vs Temperature	88
3.20 Thermal Conductivity of EDTA Vs Temperature	88
3.21 Density of Methanol Vs Temperature	89
3.22 Viscosity of Methanol Vs Temperature	89
3.23 Specific Heat of Methanol Vs Temperature	90
3.24 Thermal Conductivity of Methanol Vs Temperature	90

LIST OF TABLES

Table No.	Page No.
2.1 Physical properties of Hydrazine and Hydrazine Hydrate	3
4.1 Material Compatibility for Hydrazine Hydrate	17
5.1 Design of Storage Tank	19
5.2 Design of Stirred Vessel	21
5.3 Sizing of Crystallizer	33

NOMENCLATURE

HH – Hydrazine Hydrate

MEK – Methyl Ethyl Ketone

CAS – Chemical Abstract Species

COC – Cleveland open cup

MOC – Material of Construction

P_c – Critical Pressure, MPa

T_c – Critical Temperature, $^{\circ}\text{C}$

n_{25} – Refractive Index

ρ – Density

μ, μ_T – Viscosity

C_p – Specific Heat

K – Thermal Conductivity

ρ_B – Density of ball used, kg/m^3

ρ_f – Density of fluid

t – Time

H – Height or Length

D – Diameter

m – Mass flow rate, kg/hr

N – Speed of agitator

D_I – Diameter of impeller

N_b – Power number

X_f – Feed Composition

x_d – Distillate composition

x_w – Residue composition

λ – Latent Heat of Vaporization

R_m – Minimum reflux ratio

R – Actual reflux ratio

F_{LV} – Flooding velocity

ρ_G – Density of gas

n_h – No. of Holes in pipe type distributor

d_h – Diameter of the hole in distributor

h – Height of slot in riser of packing support, mm

a – Width of slot in riser of packing support, mm

d_r – Diameter of riser of packing support, mm

n – Number of risers

Q – Volumetric Flow Rate

A – Cross Sectional Area

V – Velocity of liquid

N_{Re} – Reynolds Number

f – Friction factor

Sp. Gr – Specific Gravity

1 INTRODUCTION:

1.1 System Considered:

The system considered for study, is a hydrazine hydrate production plant. The process is, patented by Indian Institute of chemical technology based on peroxide-ketazine route, taken as the system to design. The process description of PCUK et al. provides relevant and valuable information required for the design.

Raw materials fed to the plant are ammonia and hydrogen peroxide (may contain Methyl Ethyl Ketone as a catalyst and acetamide as a activator) in which ammonia is in excess. Various unit operations and processes are required to be taken care of which is described in some detail below. The major units in the process plant are the reactor, separator and the distillation section.

1.2 Objective of the Project:

Considering the importance of the present process, work is undertaken to design the hydrazine pilot plant and scale up the reactor from bench scale to pilot scale based on laws of similitude. The objectives of the present project are the followings.

- ✓ To scale up hydrazine hydrate process from 200 gm bench scale to 12 kg/hr on pilot scale continuously. This part involves design of major equipment, ancillary equipment.
- ✓ To design the allotment process equipments such as storage tanks, stirred vessels, packed distillation tower.
- ✓ To design heat exchange equipment such as Thermosyphon reboiler, Horizontal reboiler, shell & tube heat exchanger.
- ✓ To design piping system which includes line sizing, pump selection and sizing, orifice sizing.
- ✓ To scale up CSTR from bench scale to pilot scale using geometric similarity.

2. LITERATURE REVIEW:

2.1 Hydrazine:

- ✓ It is an inorganic, colorless, highly polar, hygroscopic liquid with ammonia - like odor, and a powerful reducing agent or electron donor⁽¹⁾.
- ✓ Hydrazine is produced commercially primarily as aqueous solutions, typically 35, 51.2, 54.4, and 64 wt% N₂H₄ (54.7, 80, 85, and 100% hydrazine hydrate).
- ✓ Anhydrous hydrazine is produced for rocket propellant and limited commercial applications⁽²⁾.

2.1.1 Timeline:

- ✓ It was first prepared in 1887 by Curtius as the sulfate salt from diazoacetic ester.
- ✓ Thiele (1893) suggested that the oxidation of ammonia with hypochlorite should yield hydrazine.
- ✓ In 1906 Raschig demonstrated this process, variations of which constitute the chief commercial methods of manufacture in the 1990s.
- ✓ The first large-scale use of hydrazine was as fuel for the rocket-powered German ME-163 fighter plane during World War II.
- ✓ In 2004, world capacity is 46, 2100 metric tons, reported as N₂H₄⁽³⁾.

2.1.2 Applications:

- ✓ Hydrazine and its simple methyl and dimethyl derivatives have endothermic heats of formation and high heats of combustion. Hence, these compounds are used as rocket fuels.
- ✓ Other derivatives are used as gas generators and explosives.
- ✓ Hydrazine, a base slightly weaker than ammonia, forms a series of useful salts.
- ✓ As a strong reducing agent, hydrazine is used for corrosion control in boilers and hot-water heating systems also for metal plating, reduction of noble-metal catalysts, and hydrogenation of unsaturated bonds in organic compounds.
- ✓ Hydrazine is also an oxidizing agent under suitable conditions.
- ✓ Hydrazine is the starting material for many derivatives, among them foaming agents for plastics, antioxidants, polymers, polymer cross-linkers and chain-extendors, as well as fungicides, herbicides, plant-growth regulators and pharmaceuticals.
- ✓ Hydrazine is also a good ligand.

2.2 PROPERTIES:

2.2.1 Physical Properties⁽⁸⁾:

Table 2.1: Physical properties of hydrazine and Hydrazine Hydrate:

Property	Hydrazine	Hydrazine Hydrate
Formula	N ₂ H ₂	N ₂ H ₂ .H ₂ O
Molecular Weight	32.0453	50.0607
CAS Registry Number	302-01-2	7083-57-8
Freezing Point, °C	2.0	-51.6
Boiling Point, °C	113.5	119.4
Vapour pressure at 25 ⁰ C, kPa ^a	1.92	1.2
Critical constants		
Pc, MPa ^b	14.69	
Tc, °C	380	
dc, g/ml	0.231	
Liquid density at 25 ⁰ C, g/L	1.004	1.032
Surface tension, 25 ⁰ C, mN/m(=dyne/cm)	66.45	74.3
Liquid viscosity, 25 ⁰ C, mPa.s(=cP)	0.913	1.5
Refractive index, n ₂₅	1.4683	1.4644
Heat of vaporisation, kJ/mol ^c	39.079	47.7
Heat of fusion, kJ/mol	12.66	
Heat capacity, 25 ⁰ C, J/(g.K)	3.0778	
Heat of combustion, kJ/mol	-622.1	
Heat of formation, kJ/mol	50.434	-242.71
Free energy of formation, kJ/mol	149.24	
Entropy of formation, J/(mol.K)	121.21	
Flash point, COC ^d	52	72

^aTo convert kPa to mm Hg, multiply by 7.5

^bTo convert Mpa to atm, divide by 0.101

^cTo convert J to cal, divide by 4.184

^dCOC=Cleveland open cup

2.2.2 Chemical Properties:

Thermal Decomposition:

- ✓ Hydrazine is a high energy compound having a high positive heat of formation.
- ✓ However, elevated ($>200^{\circ}\text{C}$) temperatures are needed before appreciable decomposition occurs.
- ✓ The decomposition temperature is lowered significantly by many catalysts, particularly copper, cobalt, molybdenum, ruthenium, iridium, and their oxides.

Reductions:

- ✓ Hydrazine is a very strong reducing agent.
- ✓ In the presence of oxygen and peroxides, it yields primarily nitrogen and water with more or less ammonia and hydrazoic acid.

Metal Reductions:

- ✓ Essentially all the metals of the transition, lanthanide, and actinide series have been reduced using hydrazine to either a lower valence state or to the metal.
- ✓ The advantage of using hydrazine in these applications is that it is completely consumed and leaves no residue.
- ✓ Such reductions can be used to make silver mirrors⁽²⁾; for electroless plating of nickel^(3,4), gold⁽⁶⁾, cobalt, iron, chromium on metal or plastic⁽⁵⁾; and for the preparation of noble-metal catalysts

Carbonyl Reductions:

- ✓ The classical Wolff-Kishner reduction of ketones (qv) and aldehydes (qv) involves the intermediate formation of a hydrazone, which is then decomposed at high temperatures under basic conditions to give the methylene group, although sometimes alcohols may form⁽⁷⁾.

Hydrogenations:

- ✓ Reduction of organic compounds with hydrazine and its derivatives is covered in several reviews⁽³⁹⁾. These procedures have some advantages over conventional pressure hydrogenations in being more selective in their attack, sometimes

stereospecific (cis addition), and in not requiring the use of hydrogen gas and high pressure equipment.

Catalytic Hydrogenations:

- ✓ Nitro compounds, primarily aromatics, are reduced using hydrazine in the presence of standard hydrogenation catalysts such as Raney nickel or ruthenium on carbon. The products are generally the corresponding amines.

Alkylhydrazines:

- ✓ Mono- and higher substituted alkyl hydrazines can be made by alkylation of hydrazine using alkyl halides.

Aromatic Hydrazines:

- ✓ A general synthesis for arylhydrazines is via diazotization of aromatic amines, followed by reduction of the resulting diazonium salt ⁽⁹⁾:

Hydrazones and Azines:

- ✓ Depending on reaction conditions, hydrazines react with aldehydes and ketones to give hydrazones⁽¹⁰⁾, azines⁽¹¹⁾, and diaziridines⁽¹²⁾, the latter formerly known as isohydrazones.

Heterocyclics:

- ✓ One of the most characteristic and useful properties of hydrazine and its derivatives is the ability to form heterocyclic compounds. Numerous pharmaceuticals, pesticides, explosives, and dyes are based on these rings ⁽¹³⁾.

2.3 METHODS OF PRODUCTION

For most uses, hydrazine is produced as hydrazine hydrate in a formulation with water. The hydrate may be produced commercially by three methods:

2.3.1 Raschig Process

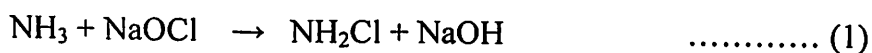
2.3.2 Urea Based Process

2.3.3 Peroxide-Ketazine Process

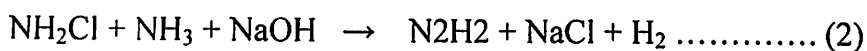
2.3.1 Raschig Process:

It basically involves oxidation ammonia with hypochlorite. The reaction mechanism is as follows ⁽²⁰⁾:

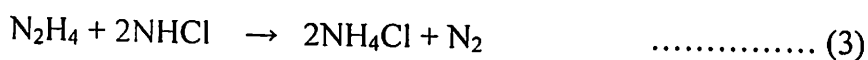
Formation of chloramines at low temperature, rapid reaction:



Excess ammonia reacts with chloramines, slow reaction at 120⁰ C to 150⁰ C under pressure:



Undesirable reaction of further oxidation hydrazine to chloramines, 18 times faster than its formation:



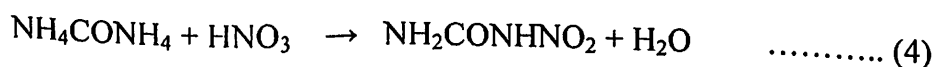
This can be avoided by presence of catalysts like metal ions, especially Cu (II).

2.3.1 Urea Based Process:

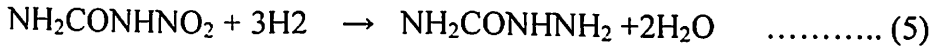
Nitration of amido compounds followed by hydrogenation and ammonolysis gives hydrazine ⁽⁸⁾:

Amido compounds such as urea, acetamide, formamide, benzamide and lauramide are usually employed for this reaction.

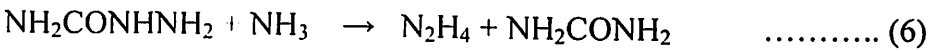
Nitro urea and water are obtained in this step:



Hydrogenation with or without catalyst like nickel at temperature between 50 °C and 400 °C and at a pressure 15 to 2000 pounds per square inch:



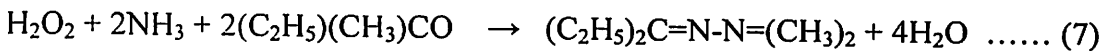
Ammonolysis at 50 °C to 200 °C and pressure 700 to 1200 pounds per square inch:



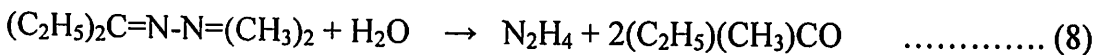
2.3.1 Peroxide-Ketazine Process:

Ammonia is oxidized by hydrogen peroxide in the presence of a ketone (Methyl Ethyl Ketone/Acetone) to form azine which on hydrolysis forms hydrazine:

A Molar ratio of $\text{H}_2\text{O}_2:\text{MEK}:\text{NH}_3=1:2:4$ is taken, acetamide and disodium hydrogen phosphate activates H_2O_2 , reaction is carried out at atmospheric pressure and temperature between 0 °C and 50 °C:



This aqueous MEK-azine is hydrolyzed under pressure (2 to 20 bar) at temperature between 150°C and 200 °C.



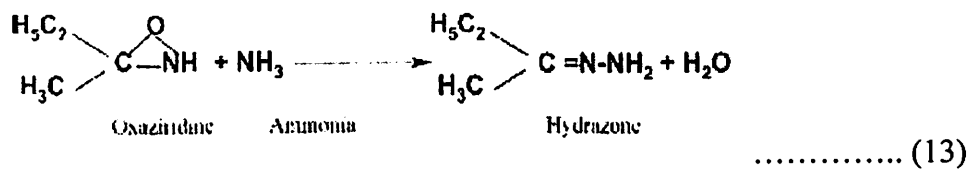
2.4 SELECTION OF PROCESS:

Production of Hydrazine is favoured via Hydrogen peroxide route due to the following reasons:

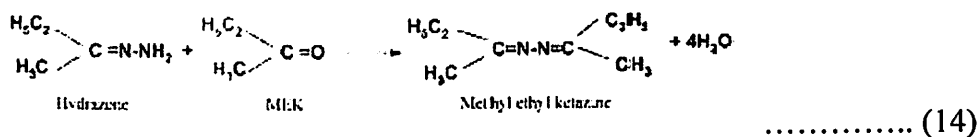
- ✓ Raw material availability of hydrogen peroxide, since it is one of the product of the client.
- ✓ Less effluent generated; hence less effluent treatment cost.
- ✓ Less raw material costs(Nikitha et. al., 2010)
Peroxide: Urea based: Raschig = 1: 2.2 : 1.25
- ✓ Less utility cost (Nikitha et. al., 2010)
Peroxide: Urea based: Raschig = 1:1.05:1.04
- ✓ Minimum Total Capital Investment (Nikitha et. al., 2010)
Peroxide: Urea based: Raschig = 1: 1.387:1.48
- ✓ Maximum rate of return (Nikitha et. al., 2010)
Peroxide: Urea based: Raschig = 59:23.6:14
- ✓ Minimum Payback period (Nikitha et. al., 2010)
Peroxide: Urea based: Raschig (yr.) = 3.29:13.1:5.5
- ✓ Minimum Break-Even Point (Nikitha et. al., 2010)
Peroxide: Urea based: Raschig (% Production) = 42:55:75

This above reasons justifies that the peroxide-ketazine process to produce Hydrazine hydrate is not only profitable but also has lesser impact on environment when compared with other methods of process.

The oxaziridine oxidizes a second molecule of ammonia to form hydrazone:



Hydrazone with excess ketone forms azine:



This MEK-azine forms an immiscible upper organic layer which can be easily removed by decantation. The lower, acetamide and sodium phosphate is concentrated to remove water and then recycled to the reactor after purging of water-soluble impurities.

2.6 PROCESS SYNTHESIS:

The final scope project is to produce 8000 tons per annum of Hydrazine Hydrate. This can be achieved on continuous basis. Since the reaction kinetics is not well studied, it is necessary to understand the behavior of the process at bench scale. The bench scale studies were carried out for the production of Hydrazine hydrate at 200 gm batch. This resulted in employing reactive distillation in the process, which necessitated the demonstration of the process at pilot scale on continuous basis. The design basis for the pilot scale was selected to be 12 kg per hour continuous.

The following are the advantages that can be achieved by employing reactive distillation in the process:

Advantages over Reactor step:

- ✓ Heat of reaction is utilized in distillation for boiling duty
- ✓ Continuous removal of the product at process conditions increases equilibrium condition and converts reversible reaction into irreversible.
- ✓ It provides high conversion compared to conventional reaction step.

Advantages over Distillation step:

- ✓ Reduction of capital cost as separate equipment for distillation is not required; also pump, piping and instrumentation can be eliminated.
- ✓ Reduced reboiler duty; since heat of reaction can be utilized.
- ✓ As conversion will be higher, lesser number of stages for same values of reflux; lesser size of distillation column; less amount of reflux also decreases sizes of condenser and reboiler.
- ✓ Operating cost is reduced; since recycling of the reactants can be reduced considerably.

3. DESIGN DATA ESTIMATION

3.1 *Properties Estimation of the Components*

Properties to be estimated are as follows:

- ✓ Density
- ✓ Viscosity
- ✓ Specific Heat
- ✓ Thermal Conductivity

Components involved in the process are as follows:

- ✓ Hydrogen Peroxide (H_2O_2)
- ✓ Methyl Ethyl Ketone
- ✓ Acetamide
- ✓ Methyl Ethyl Ketazine
- ✓ Water (H_2O)
- ✓ Hydrazine hydrate ($\text{N}_2\text{H}_4 \cdot \text{H}_2\text{O}$)
- ✓ EDTA
- ✓ Methanol

In this section, various properties have been evaluated experimentally, extracted from Aspen Properties® and from literature cited. Some properties have been checked with group contribution techniques. Temperature dependent properties have been evaluated for the whole range of the component being used.

See APPENDIX 4: From figure 3.1 to figure 3.24

Methyl Ethyl ketazine, being an intermediate, is not available in Aspen Properties as well as Hydrazine hydrate. Therefore, Properties have been experimentally evaluated. Following apparatus has been used to measure the properties:

- ✓ For density determination, specific gravity bottle have used and the sample is collected after from the range of room temperature to 180⁰C.
- ✓ Hopler's apparatus has been used to find the viscosity in which the apparatus is maintained at the desired temperature and a ball of known density and measuring constant value "k" is allowed to pass through the fluid. The is noted and viscosity is measured using the following correlation.

$$\mu_T = k(\rho_{ball} - \rho_{fluid})_T \cdot t$$

Where μ_T is the viscosity, kg/m.s at medium temperature T (⁰C)

$$k = 1.1345E-03$$

ρ_{ball} = Density of ball used, kg/m³(for eg. 13232 kg/m³ for metal ball)

ρ_{fluid} = Density of fluid at temperature maintained T (⁰C)

t = time taken by ball to travel through the column of fluid, sec.

- ✓ Specific heat and thermal conductivity measurements have been outsourced from other laboratory due to non-working condition of the Parr calorimeter.

3.7 Densities and Viscosities of Methyl Ethyl Ketazine and Hydrazine Hydrate:

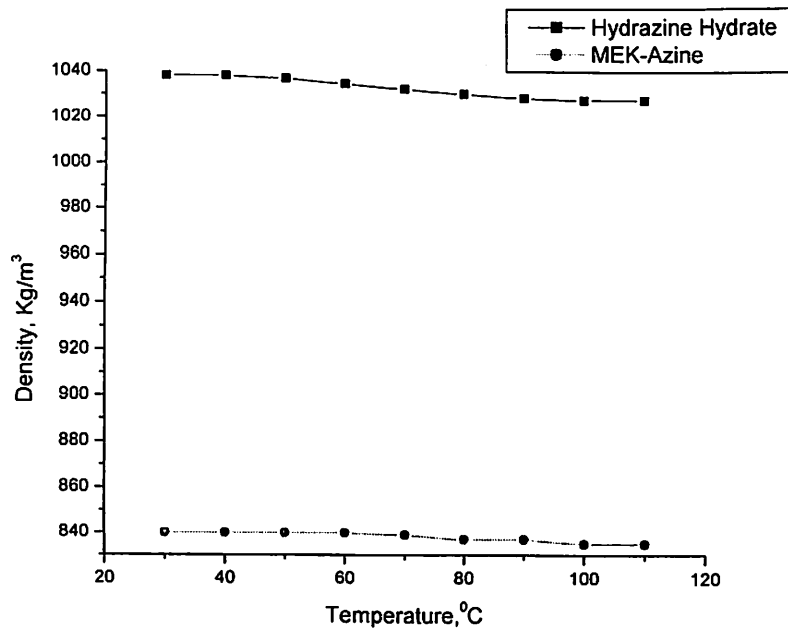
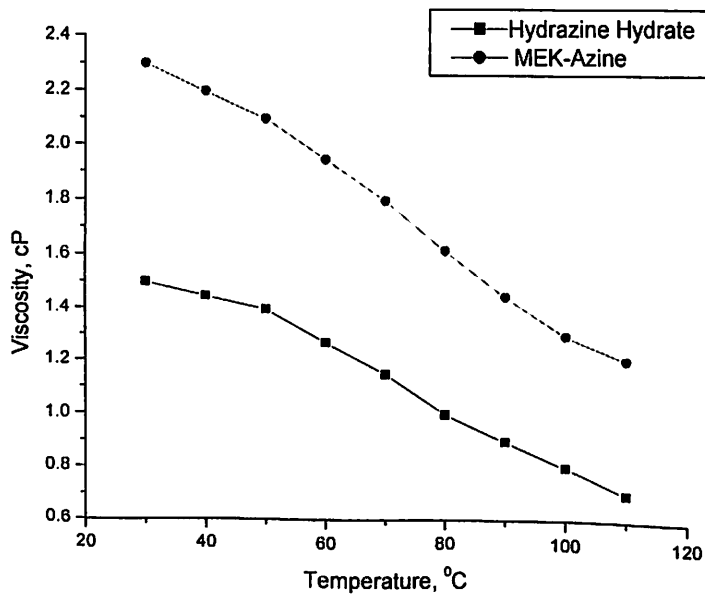


Fig.3.25 Densities of MEK-Azine and HH Vs Temperature



3.26 Viscosities of MEK-Azine and HH Vs Temperature

Heat Capacity of MEK-Azine:

Heat capacity, C_p = 2.3 J/g^{°C} (0.077 Kcal / mol. K) At 150 °C
 = 2.17 J/g^{°C} (0.0726 Kcal / mol. K) at 30 °C

4. PROCESS DESIGN ASPECTS:

4.1 Material of Construction Philosophy:

In choosing the proper materials of construction for hydrazine and others reactants involved, it is necessary to consider both the effects of the material on the stability and quality of the hydrazine as well as the effect of the hydrazine on the material of construction. Hydrazine is thermally stable, storable for years without adverse effects either to the product or the storage container provided the recommended materials are used, all systems are clean, and an inert gas, i.e., nitrogen, is maintained over the system at all times. Table 2 is a brief listing of materials compatibility⁽¹²⁵⁾.

Table 4.1: Material Compatibility for Hydrazine Hydrate:

Material	Hydrazine Concentration, wt% N ₂ H ₄			
	<10	35	54.4	64
Stainless Steel				
304L	S	S	S	S
347	S	S	S	S
316 ^b	S	S	S	S
Cold-rolled steel	S	NR	NR	NR
Copper	NS	NS	NS	NS
Brass	NS	NS	NS	NS
Aluminium	NS	NS	NS	NS
PTFE ^c	S	S	S	S
Polyethylene	S	S	S	S
Polypropylene	S	S	S	S

^aS = generally satisfactory; NR=not recommended; NS=not suitable (Decomposition)

^bUpto 65 °C; ^cPTFE = polytetrafluoroethylene

As shown, materials generally considered satisfactory for all N_2H_4 concentrations, including anhydrous, are 304 L and 347 stainless steels having <1.0 wt% molybdenum, a catalyst for the decomposition of hydrazine. For concentrations <10%, cold-rolled steel is satisfactory. Among the nonmetallic materials, poly(tetrafluoroethylene), polyethylene, and polypropylene are suitable; PVC is not recommended. Ethylene-propylene-diene monomer (EPDM) rubber ⁽¹²⁶⁾ and polyketones and polyphenylene sulfides ⁽¹²⁷⁾ are reportedly suitable for use with anhydrous hydrazine. Many factors are involved in materials compatibility; a final choice may require some testing. For example, low (<250 ppm) concentrations of CO_2 in anhydrous hydrazine accelerate the decomposition of hydrazine in stainless steel ⁽¹²⁸⁾. Extensive information and bibliographies are available on suitable materials of construction in (7,130).

5. EQUIPMENT DESIGN:

5.1 Storage Tanks:

5.1 Sizing of the Storage Tanks:

In all sections, total number of storage tanks is 54 including day tanks, intermediate tanks, recycle tanks etc. These have been sized in accordance with IS-4179. Volume of the tank has been fixed in terms of liters and H:D is taken as 1.25.

5.1.1 ST – 101:

Duty: Hydrogen peroxide Day Tank

Sizing:

Total Hydrogen Peroxide required for a day = 135.45 kg = 104.2 lit

Designing for 3 days = 312.6 lit

Table 5.1 Design of Storage Tank

Type	Cylindrical with Dished ends
H/D ratio	1.25
Nominal Capacity, lit	630
Diameter, mm	800
Height, mm	1100
Aggregate Volume (Kr = 0.06D)	665
Free board, %	52.3
<i>Cylindrical Shell</i>	
Volumetric Capacity/m. of Height, lit	502
Surface/m. of Height, m ²	2.51
<i>Torispherical Head</i>	
Volumetric Capacity/ m. of Height, lit	56.8
Surface, m ²	0.78
Liquid height= $\frac{\text{Act.Vol.}-\text{Vol.Cap.}(Tori.Head)}{\text{Vol.Cap.}(Shell)}$,m	0.51

Design Condition:

Design Temperature, °C	25/35
Design Pressure, atm	1

NOZZLE SIZING:

Inlet for H₂O₂ (N1):

Volume of MeOH = 312.6 lit

Charging time = 30 min

Flow rate = 10.4 lpm

Pumped flow-Discharged Flow, Velocity – 4 ft/sec, Nozzle size – 25.44 mm (From Charts[#])

Outlet for H₂O₂ (N2):

Volume of H₂O₂ Solution = 104 lit/day

Discharging time = 15 min

Flow rate = 6.94 lpm

Pumped Flow - Suction, Velocity – 2 ft/sec, Nozzle size – 25.44 mm

Other Nozzles:

Vent = 25.44 mm

Spare = 25.44 mm

Hand Hole/Side glass = 200/100 mm

Level Gauge = 20 mm

5.2 Design and Sizing Stirred Vessels:

5.2.1 SV-101:

Duty: Dissolution Acetamide Batch tank

Sizing:

Table 5.2 Design of Stirred Vessel

Type	Cylindrical with Dished ends
H/D ratio	1.25
Nominal Capacity, lit	630
Diameter, mm	800
Height, mm	1100
Aggregate Volume ($K_r = 0.1D$)	665
Free board, %	28.3
<i>Cylindrical Shell</i>	
Volumetric Capacity/m. of Height, lit	502
Surface/m. of Height, m ²	2.51
<i>Torispherical Head</i>	
Volumetric Capacity/m. of height, lit	56.8
Surface, m ²	0.78
Liquid height = $\frac{Act.Vol.-Vol.Cap.(Tori.Head)}{Vol.Cap.(Shell)}$, m	0.836
Material of Construction	MS

Baffle Design:

Table 5.2 Design of Stirred Vessel (Contd.)

Baffle Height, m	$1.1 \times 0.836 = 0.9196$
Baffle width, mm	66

Design Condition:

Table 5.2 Design of Stirred Vessel (Contd.)

Design Temperature, °C	65/75
Design Pressure, atm	1
Batch Time, min	60
Stirring Time, min	60
Discharging, min	60

Jacket Design:

Heat of Solution = 9791 kcal/kg

Cooling Time = 60 min

Cooling water requirement:

$$Q = m C_p \Delta T$$

$$9791 = m \times 1 \times 5$$

$$m = 1958.5 \text{ kg/hr}$$

Temperature of Methonolic Acetamide due to heat of solution

$$Q = m C_p \Delta T$$

$$9791 = 405.2 \times 0.59 \times (T - 30)$$

$$T = 70.95^\circ\text{C}$$

$$\Delta T = \frac{(71 - 30) - (40 - 35)}{\ln \left[\frac{(71 - 30)}{(40 - 35)} \right]} = 17.11$$

For Stirred Tank, Jacketed Water-liquid, $U = 25 - 60 \text{ Btu/hr.ft}^2.\text{ }^\circ\text{F}$

$$\text{Heat Transfer Area} = \frac{Q}{U \Delta T_{\ln}} = \frac{9791}{50 \times 4.88 \times 17.11} = 2.345 \text{ m}^2$$

Jacket height = 725 mm

Surface / m. of Ht. = 2.51 m²

Total heat transfer area provided = 2.51 × 0.727 + 0.78 = 2.59 m²

$$\text{Excess area} = \frac{2.59 - 2.345}{2.345} = 10.44 \%$$

AGITATOR DESIGN:

$$\text{Diameter of impeller (Di)} = \frac{1}{3} \times \text{Tank Diameter} = \frac{1}{3} \times 800 = 270\text{mm}$$

$$\text{Speed (N)} = 100 \text{ rpm} = 1.667 \text{ rps}$$

$$N_{re} = \frac{\rho N D_i^2}{\mu} = \frac{938 \times 1.667 \times 0.27^2}{0.01012} = 11,263$$

From power curves,

$$\text{Power number (Nb)} = 1.4,$$

For Stainless steel, six pitched blade turbine, 4 baffled

$$\text{Power} = \frac{N_p \rho N^3 D_i^5}{g_c} = \frac{1.5 \times 938 \times 1.67^3 \times 0.27^5}{1} = 0.012 \text{ hp}$$

Recommended – 0.5 hp

NOZZLE SIZING:

Inlet for Water (N1):

Volume of MeOH = 455.4 lit

Charging time = 15 min

Flow rate = 30.56 lpm

Gravity Flow, Velocity – 0.5 ft/sec, Nozzle size – 80 mm (From Charts[#])

Inlet for Acetamide (N2):

Weight of Acetamide = 455.4 kg

Hopper Size = 80 mm

Outlet for Acetamide Solution (N3):

Volume of Acetamide Solution = 455.4 lit

Discharging time = 15 min

Flow rate = 30.56 lpm

Pumped Flow - Suction, Velocity – 2 ft/sec, Nozzle size – 40 mm

Vent = 25 mm

Spare = 25 mm

Cooling Water inlet = 40

Cooling Water outlet = 40

Hand Hole/Side glass = 200/100

Stirrer shaft inlet = 50 mm

Thermowell = 25 mm

5.3 Distillation Column Design (Packed Tower-T-304):

Feed Condition:

System: Methanol-Water

Mole Fraction: 87 wt% of Methanol

Flow Rate: 2170 kg/day

Temperature: 30⁰C

Product Requirement:

Top Product: Methanol, 99.9 wt%

Bottom Product: Water, 99 wt%

Assumptions:

- ✓ Operating Pressure at 1.1 atm to account for pressure drop in the column.
- ✓ Molal heat of vaporization is constant
- ✓ No heat losses
- ✓ No heat if mixing

Calculation:

Feed Composition, $x_f=0.79$

Distillate composition, $x_d=0.997$

Residue composition, $x_w=1-0.996=0.004$

Feed temperature, $T = 30^0\text{C} = 303.15\text{K}$

Boiling point of Methanol = 65⁰ C

Boiling point of Water = 100⁰ C

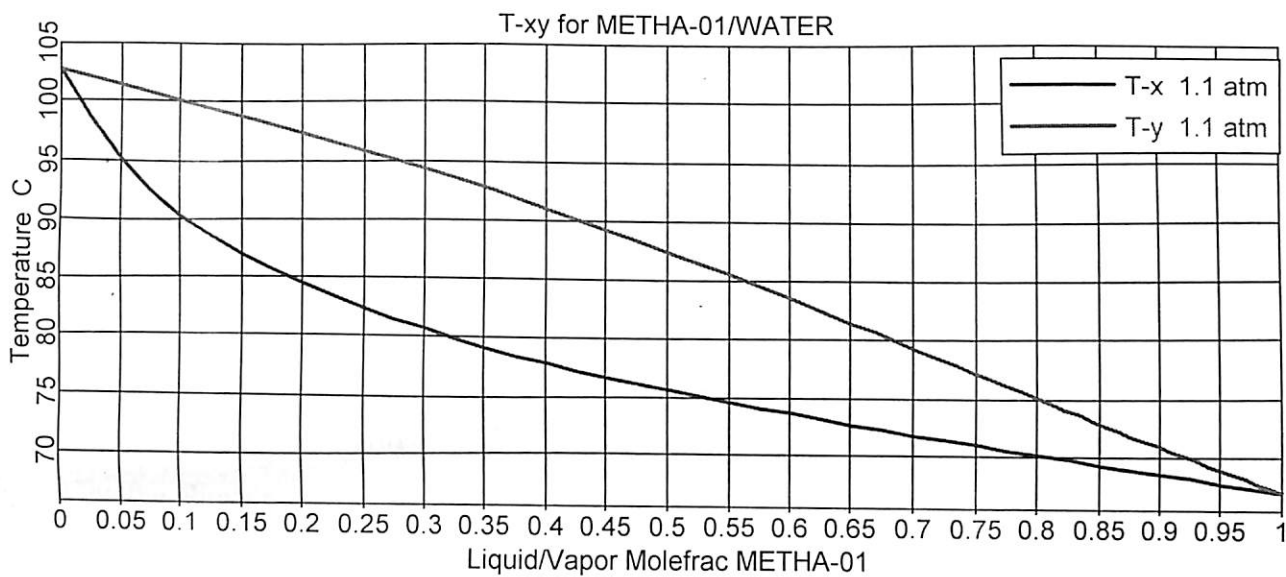
Average boiling point of the system = $(0.79 \times 65) + (0.31 \times 100) = 82.35^0\text{C}$ (Assumption)

From T-xy diagram,

For $X_f = 0.79$,

Bubble Point = 70.5⁰C

Average temperature = $\frac{30+70.5}{2} = 50.25^0\text{C}$



5.1 T-xy Diagram for T-304

	C_p at 50.25°C , KJ/Kmol-K	λ at 70.5°C , KJ/Kmol-K
Methanol	84.31	34800
Water	75.359	40600

Average molar heat capacity of feed,

$$C_{p_{\text{feed}}} = 0.79 \times 84.31 + 0.31 \times 75.359 = 89.9662 \text{ KJ/Kmol-K}$$

Average latent heat of vaporization,

$$\lambda_{\text{feed}} = 0.79 \times 34800 + 0.31 \times 40600 = 40078 \text{ KJ/Kmol}$$

$$q = \frac{\text{Heat to vaporize 1 mol of feed}}{\text{Molar latent heat of feed}}$$

$$q = \frac{\lambda + C_p \Delta T}{\lambda} = \frac{40078 + 89.9662 \times (50.5 - 30)}{40078} = 1.12$$

Short-Cut Method:

Calculation of minimum reflux ratio (Underwood Method):

$$\frac{R_m x_f + q x_d}{R_m(1 - x_f) + (q - 1)(1 - x_d)} = \alpha_{avg} \frac{[x_d(q - 1) + x_f(R_m + 1)]}{(R_m + 1)(1 - x_f) + (q - 1) + (1 - x_d)}$$

$$\alpha_{avg} = \frac{K_{Benzene}}{K_{Toluene}} = \frac{1.4089}{0.5918} = 3.83$$

$$\frac{0.5R_m + 1.1292 \times 0.997}{0.5R_m + 1.1292 \times 0.003} = 3.83 \frac{0.997 \times 0.1292 + 0.5(R_m + 1)}{(R_m + 1)(1 - 0.5) + (0.1292) \times (1 - 0.003)}$$

By solving the above equation results, $R_m = 4.1$

Let actual reflux ratio be 1.4 times minimum reflux ratio (1.2 - 1.5)

Therefore, Actual reflux ratio, $R = 1.4 \times 1.2972 = 4.51$

Minimum number of theoretical stages (Fenske Method):

$$N_m + 1 = \frac{\log \left[\left(\frac{x_d}{1 - x_d} \right) \left(\frac{1 - x_w}{x_w} \right) \right]}{\log(\alpha)} = \frac{\log \left[\left(\frac{0.997}{0.003} \right) \left(\frac{0.004}{0.996} \right) \right]}{\log(3.83)}$$

$$N_m = 12.0547 \cong 13 \text{ Stages}$$

Theoretical stages (Gilliland Method):

$$\frac{N - N_m}{N + 1} = 1 - \exp \left[\left(\frac{1 + 54.4X}{11 + 117.2X} \right) \left(\frac{X - 1}{X^{0.5}} \right) \right]$$

$$\text{Where } X = \frac{R - R_{min}}{R + 1} = \frac{1.81608 - 1.2972}{1.82608 + 1} = 0.1842$$

Solving the above equation results, $N = 11.13526 \cong 12 \text{ Stages}$

Rigorous Method:

McCabe Thiele Method:

This method has been coded in mapple®, it requires following parameters to generate the plot

Operating Parameters	
<i>Mole Fraction of Light Component in Feed</i>	<input type="text" value="0.79"/>
<i>Mole Fraction of Light Component in Distillate</i>	<input type="text" value="0.99"/>
<i>Mole Fraction of Light Component in Bottoms</i>	<input type="text" value="0.05"/>
<i>Reflux Ratio as a Multiple of Minimum Reflux Ratio</i>	<input type="text" value="1.5"/>
<i>Thermodynamic State of Feed (q-value)</i>	<input type="text" value="1.12"/>
<i>Relative Volatility of Binary Feed</i>	<input type="text" value="3.32"/>
<input type="button" value="Draw McCabe-Thiele Plot"/>	

Fig. 5.2 Input to Mapple Code for No. of Equilibrium Stages

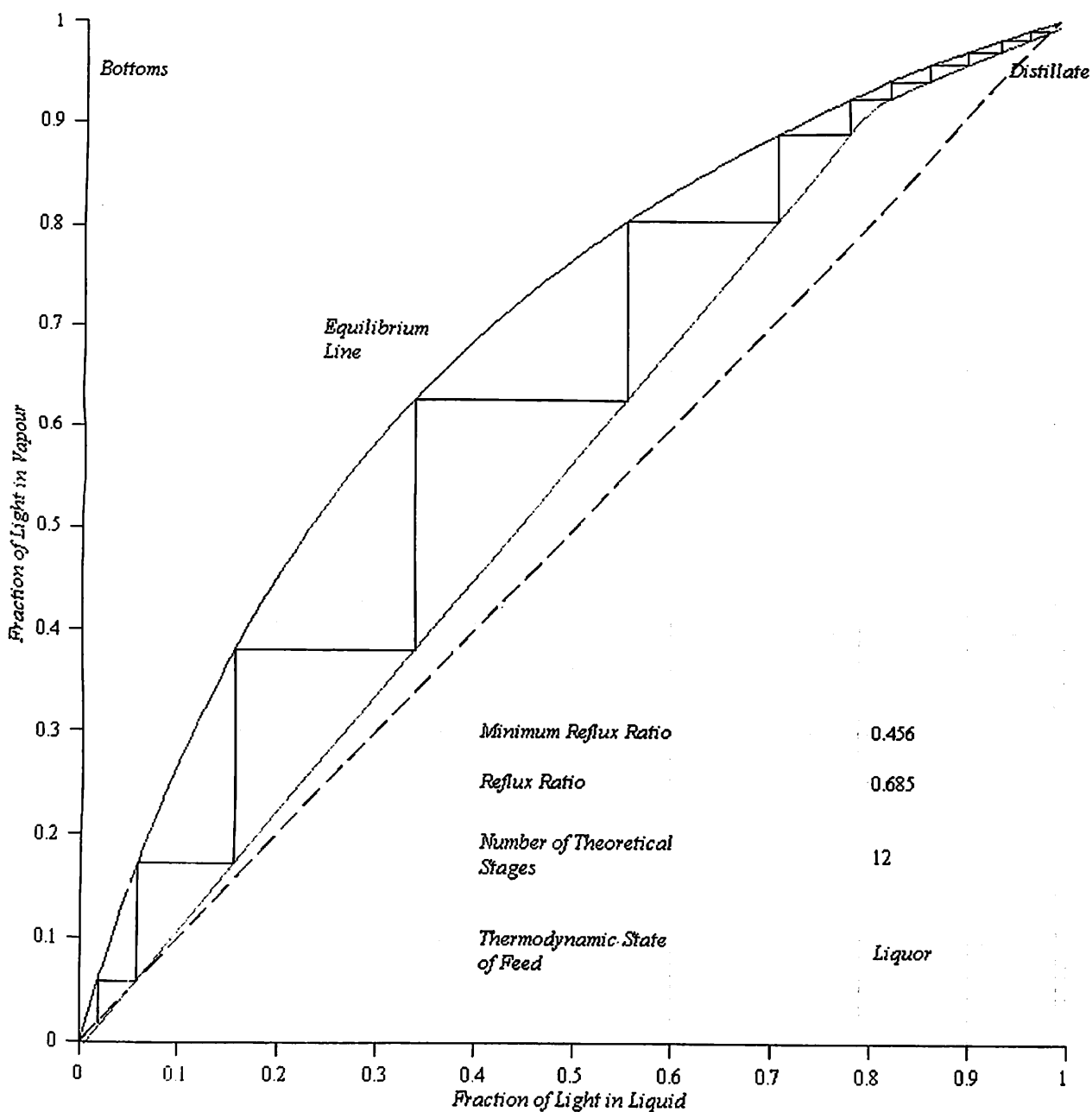


Fig. 5.3 Mc-Cabe Thiele Diagram for T-304

Design of Packed Bed for the system:

See Appendix 4

Sizing of Packed Tower:

Densities:

$$\rho_L = 0.95 \text{ g/cc} = 950 \text{ kg/m}^3$$

$$\begin{aligned}\rho_G &= \frac{PM}{RT} = \frac{1 \cdot 32}{82.06 \cdot (273 + 70)} = 1.14 \cdot 10^{-3} \text{ g/cc} \\ &= 1.14 \text{ kg/m}^3\end{aligned}$$

$$F_{LV} = \frac{L'}{G'} \sqrt{\frac{\rho_G}{\rho_L - \rho_G}} = \frac{5.982}{5.33} \sqrt{\frac{1.14}{950 - 1.14}} = 0.039$$

From Graph, Treybal, pg. no-195

$$\frac{G'^2 C_f \mu_L^{0.1} J}{\rho_g (\rho_l - \rho_g) g_c} = 0.028$$

Taking $\mu_L = 0.6 \text{ cp}$

$$= 0.6 \cdot 10^{-2} \text{ g/cm.sec}$$

$$= 0.6 \cdot 10^{-3} \text{ kg/m.sec}$$

$J=1, g_c=1$

Gas flow-rate:

$$\begin{aligned}G' &= \left[\frac{0.028 \times 1.14 \times (950 - 1.14) \times 1}{48 \times (0.6 \times 10^{-3})^{0.1} \times 1} \right] \\ &= 1.1518 \text{ kg/m}^2\text{s}\end{aligned}$$

Mass Velocity

$G=5.33 \text{ kmoles/hr}$

$$= 5.33 \times \frac{1000}{3600} = 1.48 \text{ moles/sec} = 1.48 \times 32 \text{ g/sec}$$

$$= 47.4 \frac{g}{sec} = 47.4 \times 10^{-3} kg/sec$$

$$Area = \frac{47.4 \times 10^{-3}}{1.1518} = 0.0412 \text{ m}^2$$

$$Diameter = 0.229$$

Liquid Distributor:

Type: Pipe Type

It is recommended that one liquid stream must be provided for each 194 m² area.

Therefore,

Number of holes (distributions points) required in pipe type distributor,

$$n_h = \frac{\text{cross sectional area of tower}}{194} = \frac{\left(\frac{\pi}{4}\right)(0.229)(10^4)}{194} = 6.64 \cong 4$$

Let the velocity of liquid through the pipe = 2 m/s

$$\text{Flow rate of liquid} = \frac{191}{938} \times \frac{1}{3600} = 0.0000566 \text{ m}^3/sec$$

$$d = \sqrt{\frac{0.0000566 \times 4}{2\pi}} = 0.01886 \text{ m} = 19 \text{ mm}$$

Let the velocity of liquid through the holes = 3 m/s

Let d_h be the diameter of the hole, m

$$d_h = 0.00245 \text{ m} = 2.45 \text{ mm}$$

Liquid Redistributor:

Liquid redistributor are needed every 5 to 10 times tower diameter.

Packing Support:

Packing support should be selected such that flow area provided by packing support for the flow of gas should be greater than flow area provided by packing material.

Void percentage for 25 mm size SS Pall rings = 92 %

Hence with Pall rings, any gas-injection type packing support must be provided. Let the type of packing support is cap type packing support.

Actual outer diameter of packing support is greater than 230 mm as some portion of packing support is sandwiched between two flanges.

Let h = Height of slot in riser, mm

a = Width of slot in riser, mm

d_r = Diameter of riser, mm

n = Number of risers, mm

D_i = Inside diameter of column, mm

$$\text{Let } d_r = \frac{230}{6} = 38.33 \text{ mm}$$

Number of risers, $n = 9$

Total area of slots of risers = $n_s n h a$, where 'a' is the distance between two successive slots.

Let $a = 5$

$$n_s = \frac{\pi d_r}{2a} = \frac{\pi \times 40}{2 \times 5} = 1.25$$

Let $n_s = 2$

Hence, total area of slots of risers = $2 \times 9 \times h \times 5 \geq 0.92 \times \frac{\pi}{4} (230)^2$

$$h \geq 42.4 \text{ mm}$$

Therefore, Height of slot in riser should be atleast 42.2 mm

5.4 Crystallizer (Cr-301):

Duty: To crystallize distillate separated from organic layer for acetamide recycle (Recovery)

Total input of Water, MEK, Amide, and Ketazine = 17.986 kg

For 8 hours of operation, 143.872 kg/hr

Volume of the material = 171.27 lit

Including Vapour space (30 %) = 244.68 lit

Sizing:

Table 5.3 Sizing of Crystallizer

Type	Cylindrical with Dished ends
H/D ratio	1.25
Nominal Capacity, lit	250
Diameter, mm	600
Height, mm	775
Jacket Diameter, mm	700
Jacket Height, mm	620
Aggregate Volume (Kr = 0.1D)	266
Free board, %	8.27
<i>Cylindrical Shell</i>	
Volumetric Capacity/m. of Height, lit	218
Surface/m. of Height, m ²	2.51
<i>Torispherical Head</i>	
Volumetric Capacity/m. of height, lit	56.8
Surface, m ²	0.78
Liquid height = $\frac{Act.Vol.-Vol.Cap.(Tori.Head)}{Vol.Cap.(Shell)}$, m	0.558
Material of Construction	SS-304L

Agitator Design:

Diameter of impeller, mm ($d_i/D_t = 0.9$)	540
Height of the impeller, mm ($h_i/D_t = 0.75$)	450
Width of impeller, mm ($b_i/D_t = 0/1$)	60
Speed (N), rpm	400
Tip speed, m/s (πDN)	11.298
Reynolds' No.	3134856
Power no. (from power curves)	0.334
Power, hp	1.5
Power, hp (50% excess)	2.2

Jacket Design:

$$\text{Heat of mixing} = Q = m C_p \Delta T$$

Component	Mass, kg	C_p , kcal/kg ⁰ C
Water	3.2	1.0053
MEK	6.288	0.5991
Acetamide	11.144	0.6189
Ketazine	123.24	0.0252
	Total Heat	2072.385, kcal

$$50 \% \text{ Excess} = 3108.577 \text{ kcal}$$

Operation is for 3 hours, 1036.1925 kcal/hr

Cooling medium: 40% Ethylene glycol at -5⁰C with 2⁰C temperature approach.

$$C_p \text{ of brine at } -5^{\circ}\text{C} = 0.8129 \text{ kcal/kg}^{\circ}\text{C}$$

$$m = \frac{Q}{C_p \Delta T} = \frac{1036.195}{2 \times 0.8129} = 637.343 \text{ kg/hr}$$

NOZZLE SIZING:

Inlet for Crystallizer (N1):

Volume of Content = 171.127 lit

Charging time = 10 min

Flow rate = 17.127 lpm

50 % excess = 25.7 lpm

$$\text{Flow rate} = \frac{25.6 \times 10^{-3}}{60} = 4.284 \times 10^{-4} \text{ m}^3/\text{sec}$$

Pumped flow, Velocity -4 ft/sec = 1.2192 m/sec, Nozzle size - 25 mm (From Charts[#])

Also,

$$\text{Area} = 4.284 \times 10^{-4} / 1.21 = 3.512 \times 10^{-4} \text{ m}^2$$

Diameter = 21.4 mm (1 inch nozzle)

Outlet Nozzle (N2):

Filerate + Salt = 153.63 lp in 10 minutes + 14.489 lit = 15.36 lpm

50 % excess = 23.04

Gravity flow, Velocity 0.5 ft/sec = 0.1524 m/sec

$$\text{Area} = 0.002519 \text{ m}^2$$

Diameter = 56.6 mm

Taking 80 mm nozzle (2½ inch nozzle)

Jacket inlet Nozzle (N3):

Density of Brine = 1061.1 kg/m^3

Volumetric flow rate = $(637.343/1061.1) = 0.6006 \text{ m}^3/\text{hr} = 10.01 \text{ lpm}$

Pumped discharge, Velocity = $4 \text{ ft/sec} = 1.219 \text{ m/s}$

Area = $2.05 \times 10^{-4} \text{ m}^2$

Diameter = 16 mm

Vent = 25 mm

Spare = 25 mm

Brine inlet = 25 mm

Brine outlet = 25 mm

Hand Hole/Side glass = 200/100 mm

Stirrer shaft inlet = 50 mm

Thermowell = 25 mm

Baffle:

Type: Spiral type baffle

Baffle Pitch = 50 mm

6. PROCESS DESIGN OF PIPING SYSTEM:

6.1 Line Sizing:

6.1.1 Liquid Line Sizing:

Procedure followed in sizing the liquid lines is as follows:

✓ *A standard diameter of line is assumed (D)*

✓ *Calculation of Velocity:*

Volumetric Flow Rate (Q) —

Cross Sectional Area (A) —

Velocity of liquid (V) —

✓ *Calculation of Reynolds Number:*

Reynolds Number (NRe) —

✓ *Calculation of Friction Factor:*

If $Re \leq 2000$

Moody Friction Factor —

If $Re > 2000$

$$\frac{(\epsilon/D)^{1.1098}}{2.3257} + \left(\frac{1.149}{Re}\right)^{0.8981}$$

Fanning friction factor (f) can be calculated from Chen's formula as follows:

$$\frac{1}{\sqrt{f}} = -4 \log_{10} \left(\frac{\epsilon/D}{3.7065} + \frac{5.0452}{Re} \log_{10}(44) \right)$$

Moody Friction Factor

✓ *Pressure Drop Calculation:*

Where Sp. Gr = Specific Gravity

D = Diameter in cm

f = Moody friction factor

Q = Volumetric Flow Rate in m³/hr

ΔP = Pressure Drop in kg/cm²/km

✓ **Condition Check:**

If Pressure drop (ΔP) and velocity (V) are not within the given conditions of maximum value, then go to step 1, else stop

Line sizing has been done in two sections. Viz. Section-100 and Section-200. These calculations were done using the spreadsheet whose pictorial is found in Appendix 4.

Liquid Line Sizing example:

Line Number: 103

Duty: Hydrogen peroxide feed line

Material = 69.24 lit for 8 hrs

Mass flow rate = 8.655 lit/hr = 7.868 kg/hr

Properties required:

Density = 1.1 g/cc at average temperature range from 25⁰C to 60⁰C.

Viscosity = 1.243 at average temperature range from 25⁰C to 60⁰C.

Volumetric flow rate = 0.00649 m³/hr = 0.00649 × 1.05 = 0.006815 m³/hr

Absolute roughness = 0.04572 mm (for Stainless Steel)

Maximum velocity = 1.2-2 m/s (Recommended)

Maximum Allowable Pressure Drop = 8.8 kg/cm²/km (Recommended)

1. A standard diameter of line is assumed (D)

$$D = 0.02 \text{ m}$$

2. Calculation of Velocity:

$$\text{Cross Sectional Area (A)} = \frac{\pi}{4} D^2 = 0.0000366 \text{ m}^2$$

$$\text{Velocity of liquid (V)} = \frac{Q}{A} = 0.05 \text{ m/s}$$

3. Calculation of Reynolds Number:

$$\text{Reynolds Number (NRe)} = \frac{DV\rho}{\mu} = 345$$

4. Calculation of Friction Factor:

$$\text{Re} < 2000$$

$$\text{Moody Friction Factor} = \frac{64}{Nre} = 0.186$$

5. Pressure Drop Calculation:

$$\Delta P = 6370 \times \frac{Q^2 f}{D^2 (\text{Sp. Gr})}$$

6. Condition Check: Satisfied

The obtained value of diameter is 0.4 inch, selecting the nearest standard

Selecting 1/2 inch Sch 40, 16 BWG Pipe

6.1.2 Vapour Line Sizing:

Vapor Line sizing has also been done in a similar way

Liquid Line Sizing example:

Line Number: 109

Duty: Ammonia feed line to the scrubber

Mass flow rate = 11.31 kg/hr

Properties required:

Density = 0.66 kg/m^3 at average temperature range from 25°C to 60°C .

Viscosity = 0.00098 Poise at average temperature range from 25°C to 60°C .

Volumetric flow rate = $17.47 \text{ m}^3/\text{hr} = 17.47 \times 1.05 = 18.34 \text{ m}^3/\text{hr}$

Absolute roughness = 0.04572 mm (for Stainless Steel)

Maximum velocity = 10 m/s (Recommended)

Maximum Allowable Pressure Drop = $8 \text{ kg/cm}^2/\text{km}$ (Recommended)

1. A standard diameter of line is assumed (D)

$D = 0.026 \text{ m}$; Nominal Diameter = 1 inch

2. Calculation of Velocity:

$$\text{Cross Sectional Area (A)} = \frac{\pi}{4} D^2 = 0.000557 \text{ m}^2$$

$$\text{Velocity of liquid (V)} = \frac{Q}{A} = 9.14 \text{ m/s}$$

3. Calculation of Reynolds Number:

$$\text{Reynolds Number (NRe)} = \frac{DV\rho}{\mu} = 16,405$$

4. Calculation of Friction Factor:

Re > 2000

$$\frac{(\epsilon/D)^{1.1098}}{2.8257} + \left(\frac{7.149}{Re}\right)^{0.0701} = 0.0012$$

Fanning friction factor (f) can be calculated from Chen's formula as follows:

$$\frac{1}{\sqrt{f}} = -4 \log_{10} \left(\frac{\epsilon/D}{3.7065} + \frac{5.0452}{Re} \log_{10}(44) \right) = 11.47$$

$$\text{Moody Friction Factor} = 4(0.00759) = 0.0303$$

5. Pressure Drop Calculation:

$$\text{—————} \quad \text{kg/cm}^2/\text{km}$$

6. Condition Check: Satisfied

The obtained value of diameter is 1.315 inch, selecting the nearest standard

Selecting 1½ inch Sch 40, 16 BWG Pipe

6.2 Orifice Sizing:

Orifice meter are used in the process to due its advantages over other flow meter. Sizing here means selection of type of orifice, determination of diameter of orifice, coefficient of discharge, selection of pressure taps, finding a suitable DP transmitter.

Procedure followed in sizing the liquid orifice is as follows:

- ✓ Selection of type of orifice.

Square edged, sharp edged. For liquid, it is recommended to use square edged.

- ✓ Sizing of orifice:

Usual practice is half the diameter of pipe is taken as diameter of orifice.

- ✓ Pressure taps:

There are five standard pressure taps:

- i. Corner taps
- ii. Flange taps
- iii. Radius taps
- iv. Vena contracta taps
- v. Pipe taps

Radius taps are selected as it gives good pressure drop, higher the pressure drop more accurate the measurement of flow.

- ✓ Coefficient of Discharge:

For high turbulent flow, it is taken to be in between 0.595 to 0.62 for vena contracta, 0.595 to 0.8 for radius trap and 0.62 for corner taps.

This can also be estimated from Stolz equation as follow:

$$C_o = 0.5959 + 0.0312\beta^{2.1} - 0.184\beta^8 + 0.0029\beta^{2.5}(10^6/NRe)^{0.75} \\ + 0.91L_1\beta^4(1 - \beta^4)^{-1} - 0.0337L_2(1 - \beta^3)$$

Where $\beta = \frac{\text{Diameter of orifice}}{\text{Diameter of Pipe}}$

6.2.1 Liquid Orifice Sizing example:

INDIAN INSTITUTE OF CHEMICAL TECHNOLOGY			
Design of Orifice Meter-Liquid			
Prepared by:	Ibrahim Khan	Orifice Tag No:	O-101
Checked by:	M Pradeep Kumar; Naveen Kumar	Job No:	170000
Date:		Client:	GACL
		Project:	Hydrazine Hydrate

INPUTS			RESULTS		
Name of the Fluid	MEK-azine		Quantity	Value	Units
Quantity	Value	Units	Orifice Diameter	25	mm
Flow rate	1500	kg/hr	Type	Circular	
Pipe ID	50	mm	Edges	Square	
SCH	40		Radius Taps	Yes	
Temperature, T	32	deg C			
Density at T	995.026	kg/cu.m	Coefficient of Discharge	0.639402	
Viscosity at T	0.765	cP	Pressure Drop	0.084635	m WC
Manometric Fluid	Mercury		Manometer Reading	6.758696	mm HG
Manometric Fluid Dens:	13516.7	kg/cu.m	DP Transmitter	169.269	mm WC

Fig. 6.1 Liquid Orifice Sizing

6.2.1 Vapour Orifice Sizing example:

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Design of Orifice Meter-Gas			
Prepared by:	Ibrahim Khan	Orifice Tag No:	O-102
Checked by:	M Pradeep Kumar; Naveen Kumar	Job No:	170000
Date:		Client:	GACL
		Project:	Hydrazine Hydrate

INPUTS			RESULTS		
Name of the Fluid	NH3		Quantity	Value	Units
Quantity	Value	Units	Orifice Diameter	25	mm
Flow rate	219	Nm ³ /hr	Type	Circular	
Pipe ID	75	mm	Edges	Square	
SCH	40		Corners Traps	Yes	
Pressure	1.2	atm a	Flow rate	151.877	kg/hr
Temperature, T	35	deg C	Coefficient of Discharge	0.612833	
Viscosity at T	0.0145	cP	Pressure Drop, del P1	0.262064	m WC
Specific Ratio for gas	1.355		Pressure Drop, del P2	0.232864	mm HG
Mol. Wt. of Gas	17.03		DP Transmitter	308.31	mm WC

Fig. 6.2 Vapor Orifice Sizing

6.3 Selection of Pump:

Type of pump based on the duty is selected for the process. Capacity has to be provided along with material of construction.

Example: P-101:

Type: Injection Pump

Service: Charging H₂O₂ from feed tank to the reactor

Volume of H₂O₂ = 312.4 lit

Time of Charging = 30 min

Pump capacity = 10.4 lpm

Design Capacity = $1.5 * 10.41 = 15.62$ lpm

Minimum: 10 lpm

Maximum: 20 lpm

Temperature: Ambient

Pressure: Atmospheric

7. HEAT EXCHAGER DESIGN :

7.1 Horizontal Condenser -Thermal Design:

Algorithm for Design is as follows:

Design of Horizontal Condenser:

Heat Duty calculations:

$$Q = Q_c + Q_{\text{sub}} = \dot{m}\lambda + mC_p\Delta T$$

Where, $Q_c = \dot{m}\lambda$ =Heat duty for condensation

$$Q_{\text{sub}} = mC_p\Delta T = \text{Heat duty for subcooling}$$

λ = Latent heat of vaporization of at condensation temperature

Energy Balance:

$$Q = m_w C_{pw} (T_{\text{out}} - 32)$$

Fixing the outlet temperature of cooling water as 40°C

Mass flow rate of cooling water required

$$m_w = \frac{Q}{C_{pw}\Delta T} \text{ kg/s}$$

Let overall heat transfer coefficients be

For condensation $U_c = 800 \text{ W/(m}^2\text{.}^\circ\text{C)}$

For subcooling $U_{\text{sub}} = 200 \text{ W/(m}^2\text{.}^\circ\text{C)}$

Assuming that entire flow of cooling water is first utilized for subcooling and then for condensation.

$$Q_{\text{Sub}} = m_w C_{pw} (T' - 32)$$

Where T' = Intermediate temperature of cooling water

$$T' = ^\circ\text{C}$$

LMTD for condensation and LMTD for subcooling is calculated.

Area based on assumed values of U :

For condensation:

$$A_c = \frac{Q_c}{U_c \Delta T_{mc}}$$

For subcooling:

$$A_{\text{Sub}} = \frac{Q_{\text{Sub}}}{U_{\text{Sub}} \Delta T_{\text{mSub}}} =$$

$$\text{Total area } A = A_c + A_{\text{Sub}} = m^2$$

Based on the selected values of overall coefficients, area required for subcooling is less than area required for condensation. Hence to get the smaller size of heat exchanger horizontal position is selected. Ideally in such a case heat exchanger should be designed for both positions and the position which provides lesser heat transfer area should be selected.

Area provided for the first calculation

$$A = N_t \pi d_o L$$

$\frac{3}{4}$ OD (19.05 mm OD), 6 ft (1.83) long tubes is chosen

$$\text{Number of tubes, } N_t = \frac{A}{\pi d_o L} = \text{tubes}$$

Shell side fluid is clean fluid, Triangular pitch arrangement is selected.

$$P_T = 1.25, d_o = 23.8125 \text{ mm}$$

Number of tube side passes = 2

Tube bundle diameter:

$$D_b = d_o \left(\frac{N_t}{k_1} \right)^{\frac{1}{n_1}} = 19.05 \left(\frac{N_t}{k_1} \right)^{\frac{1}{n_1}} = \text{mm}$$

Select a fixed tube sheet heat exchanger.

Let clearance between shell ID and $D_b = 13 \text{ mm}$

Shell inside diameter = mm

Heat Transfer Coefficients:

Tube side coefficients:

Using Dittus-Bolter equation

$$\frac{h_i d_i}{k} = 0.023 \text{Re}^{0.8} \text{Pr}^{0.33} \left(\frac{\mu}{\mu_w} \right)^{0.14}$$

$$a_t = \frac{N_t}{N_p} \times \frac{\pi}{4} d_i^2 = \frac{111}{1} \times \frac{\pi}{4} \times (0.022098)^2 = \text{m}^2$$

(From Table 11.2, Ref. 2 for BWG tube $d_i = 15.749 \text{ mm}$)

Tube side mass velocity

$$G_t = \frac{\dot{m}}{a_t}$$

Tube side velocity,

$$u_t = \frac{G_t}{\rho} > 1 \text{ m/s}$$

To avoid the excessive fouling it is recommended to keep the velocity of water greater than 1 m/sec. If found less then increase the number of tube passes from 2 to 4.

$$D_b = 19.05 \left(\frac{N_t}{k_1} \right)^{\frac{1}{n_1}}$$

$$Re = \frac{d_i G_t}{\mu}$$

$$Pr = \frac{C_p \mu}{k}$$

Neglecting $\left(\frac{\mu}{\mu_w} \right)^{0.14}$

Thus tube side co-efficient can be evaluated.

Shell Side coefficient:

(a) *For condensation zone, h_{co} :*

Calculation for mean temperature of condensate film:

Let T_w = Tube wall temperature, $^{\circ}C$

$h_{co} = 1500 \text{ W}/(\text{m}^2 \cdot ^{\circ}C)$ (Assumed for first trial)

At steady state.

Heat transfer rate through condensate film = overall rate of heat transfer

$$h_{co} A_C (T_C - T_w) = U_C A_C (T_C - T_{av})$$

Where, T_C = Condensation temperature of the vapour on shell side, $^{\circ}C$

T_{av} = Average tube side fluid temperature of condensation zone, $^{\circ}C$

Shell side condensation with horizontal position:

$$h_{co} = 0.95k_L \left(\frac{\rho_L(\rho_L - \rho_V)g}{\mu_L \tau_h} \right)^{1/3} \cdot N_r^{-1/6}$$

$$\text{Density of vapour } \rho_V = \frac{PM}{RT} = \frac{PM}{T} \times \frac{T_{std}}{P_{std}V_{std}} =$$

$$\tau_h = \frac{W_c}{LN_t}$$

Average number of tubes in vertical rows

$$N_r = 2/3 N'_r = \frac{2}{3} \left(\frac{D_b}{P_t} \right) =$$

h_{co} is close enough to assume value of h_{co} . Hence, correction in the value of T_w is not required.

(b) For subcooling zone, h_{oSub} :

With horizontal position, subcooling is taking place via natural convection only for which reliable correlations are not available. Based on Kern's recommendation, h_{oSub} for horizontal position is 283.77 W/(m².°C)

Overall Heat Transfer Coefficient for Condensation:

$$U_{oc} = \frac{1}{\frac{1}{h_{oc}} + \frac{1}{h_{od}} + \frac{d_o \ln \left(\frac{d_o}{d_i} \right)}{2k_w} + \frac{d_o}{d_i} \left(\frac{1}{h_i} \right) + \frac{d_o}{d_i} \frac{1}{h_{id}}}$$

For organic vapors, $h_{od} = 10\,000$ W/(m².°C)

For cooling water, $h_{id} = 10\,000$ W/(m².°C)

With cooling water, stainless steel-304, is suitable material for the tube.

Thermal conductivity of SS 304 material, $k_w = 16.3$ W/(m.°C)

Heat transfer area required for condensation

$$A_c = \frac{Q_c}{U_{oc}\Delta T_{mc}}$$

Overall Heat Transfer Coefficient for Subcooling:

$$U_{oSub} = \frac{1}{\frac{1}{h_{oSub}} + \frac{1}{h_{od}} + \frac{d_o \ln\left(\frac{d_o}{d_i}\right)}{2k_w} + \frac{d_o}{d_i} \left(\frac{1}{h_i d}\right) + \frac{d_o}{d_i} \frac{1}{h_i}}$$

Heat transfer area required for subcooling:

$$A_{Sub,r} = \frac{Q_{Sub}}{U_{Sub}\Delta T_{mSub}}$$

Total heat transfer area required

$$A_{t,r} = A_{c,r} + A_{Sub,r}$$

Spreadsheets have been developed to design horizontal condenser which is shown as below:

INDIAN INSTITUTE OF CHEMICAL TECHNOLOGY

Design of Horizontal Condenser

Prepared by:	Ibrahim Khan	Condenser Tag No:	E-404
Checked by:	M. Pradeep Kumar, Naveen Kumar	Jcb No:	170000
Date:		Client:	GACL
		Project:	Hydrazine hydrate

INPUTS

Process Fluid		Cooling Medium		Condenser Geometry input	
Parameter	Value	Parameter	Value	Parameter	Value
Flow rate	158 kg/hr	Flow rate	1.546929238 kg/s	OD	19.05 mm
Cp	3.2 kJ/Kg	Cp	4.1868 kJ/Kg	Length	1 m
Density	786.1 kg/cu.m	Density	992.9 kg/cu.m	ID	15.748 mm
Viscosity	0.000311 kg/m.s	Viscosity	0.00072 kg/m.s	k1	0.319
K	0.02 W/(m.degC)	K	0.6228 W/(m.degC)	n1	2.142 From table
Latent Heat of Vap	1100.56 kJ/Kg	Latent Heat of Vap	2244 kJ/Kg	Type	Fixed tube sheet
Tcondensation	65 deg C	T1 inlet	32 deg C	Shell Clearance	23 mm
Tsubcooling	40 deg C	T2 outlet	40 deg C		
Liquid Prop. at	37.33894875 deg C	T intermediate	32.54211561 deg C		
Viscosity	0.00032 kg/m.s	Pcond	869 mmHg		
Density	805 kg/cu.m	N, SS-304	16.3 W/(m.degC)		
K	0.2 W/(m.degC)				
M.Wt	32.2				

Input n Results / Calculations / Table 1

7.1 Inputs for Spreadsheet for Design of Horizontal Condenser

RESULTS		
Quantity	Value	Units
Q,condensation	48.30235556	kW
Q,subcooling	3.511111111	kW
Q,total	51.81346667	kW
Condensation		
Area,condensation	2.113564888	sq. m
Sub Cooling		
Area,subcooling	1.00526225	sq. m
Total		
Area,total	3.118827138	sq. m
Tube side co-effi, hi	1730.284308	W/(sq.m-deg C)
Shell Side		
h,co	4062.892002	W/(sq.m-deg C)
h _o ,sub	283.77	W/(sq.m-deg C)
Overall Coefficient		
U _o ,condn	685.4458865	W/(sq.m-deg C)
U _o ,sub	211.1149949	W/(sq.m-deg C)
No of Tubes	53	
Pressure Drop, shell	197.7366031	Pa
Pressure Drop, Tube	434.5677796	Pa

7.2 Result from Spreadsheet for Design of Horizontal Condenser

7.2 Design of Vertical Thermosyphon Reboiler (Rb-401):

Determination of boiling point of MeOH:

$$\ln(P) = A - \frac{B}{(T + 273.15) - C}$$

For $A = 8.07$, $B = 1574.99$, $C = 238.87$, $P_t = P_v = 1.78 \text{ atm} = 1400 \text{ mmHg}$

$$T = 80.02^\circ\text{C}$$

Heat Duty:

$$Q = \dot{m}\lambda \times 1.1 \text{ (10 \% Heat loss)}$$

Where, λ = Latent heat of vaporization at the boiling point

Latent heat of vaporization of at $80.02^\circ\text{C} = 34040 \text{ KJ/Kmol}$

Heat duty,

$$Q = 3.1 \times 34040 = 29.31 \text{ kW}$$

$$10\% \text{ excess, } Q = 36.94 \text{ kW}$$

Let $\Delta T_m = 39.6^\circ\text{C}$. Assuming that this value of ΔT_m is less than and close to the critical temperature drop.

Average temperature of heating medium

$$T_h = 80 + 39.6 = 119.6^\circ\text{C}$$

From steam tables, for the steam temperature, $t_s = 120^\circ\text{C}$, saturation steam pressure = 510 kPa and latent heat of condensation of steam $\lambda_s = 1548.67 \text{ KJ/Kg}$

$$Q = \dot{m}_s \lambda_s$$

$$\dot{m}_s = \frac{36.94}{1548.67} = 0.0238 \text{ kg/hr} = 85.88 \text{ kg/hr}$$

Trial I:

Assuming overall heat coefficient, $U_o=1000 \text{ W}/(\text{m}^2 \cdot ^\circ\text{C})$

$$A = \frac{Q}{U_o \Delta T_m} = \frac{33.5 \times 1000}{1000 \times 39.6} = 0.9334 \text{ m}^2$$

$$A = N_t \pi d_o L$$

Let length of the tube $L = 1 \text{ m}$; $d_o = 25.4 \text{ mm}$

Number of tubes = 12 tubes

For 25.4 mm OD tube, 31.75 mm tube pitch ($Pt/d_o=1.25$) triangular pitch arrangement and 1-1 shell and tube exchanger.

Shell ID, $D_i = 203 \text{ mm}$ (From TEMA Sheet No.)

Recirculation ratio:

Assuming recirculation ratio = 4

Available differential head,

$$\Delta P_{av} = L(\rho_L - \rho_{avg})g$$

ρ_L = Density of liquid at B.P. = kg/m^3

Density of pentane vapour at bar and at 125°C

$$\rho_v = \frac{pM}{RT} = \frac{2 \times 27.5}{(273 + 80)} \times \frac{273}{1.01325 \times 22.414} = 1.8729 \text{ kg}/\text{m}^3$$

Average density of liquid-vapour mixture

$$\rho_{avg} = \frac{\ln(V_o/V_i)}{(V_o - V_i)}$$

V_i = Specific volume of liquid at inlet = $\frac{1}{\rho_l} = 0.001229 \text{ m}^3/\text{kg}$

For a recirculation ratio = 4

Flow rate of liquid at the outlet of reboiler

$$\dot{m}_{Lo} = 4 \times 45.395 = kg/h$$

Flow rate of vapour at the outlet of reboiler $\dot{m}_{vo} = 181.58 \text{ kg/h}$

Specific volume of vapour at outlet

$$V_o = \frac{\dot{m}_{vo}/\rho_v + \dot{m}_{Lo}/\rho_l}{\dot{m}_{vo} + \dot{m}_{Lo}} = \frac{181.58}{1.137} + \frac{45.395}{813.82} = 0.107769 \text{ m}^3/\text{kg}$$

$$\rho_{avg} = \frac{\ln(V_o/V_i)}{(V_o - V_i)} = 141.9932 \text{ kg/m}^3$$

$$\Delta P_{av} = L(\rho_L - \rho_{avg})g = 1(813.82 - 141.99)9.81 \text{ Pa} = 6590 \text{ Pa}$$

Assuming that higher sizes of the piping will be used in between base of distillation columns and thermosyphon reboiler.

Pressure loss in the system \cong Tube side pressure drop, ΔP_t

Tube side pressure drop,

$$\Delta P_t = N_p \left(8J_f \left(\frac{L}{d_i} \right) \left(\frac{\mu}{\mu_w} \right)^{-m} + 2.5 \right) \frac{\rho_{av} u_t^2}{2}$$

$$N_p = 1, \frac{\mu}{\mu_w} \cong 1, \rho_{av} = 260.14 \text{ kg/m}^3$$

Tube inside diameter

$$d_i = 22.098 \text{ mm (16 BWG tube)}$$

Tube inside flow area

$$a_t = \frac{N_t}{N_p} \times \frac{\pi}{4} d_i^2 = \frac{111}{1} \times \frac{\pi}{4} \times (0.022098)^2 = 0.004534 \text{ m}^2$$

Tube side mass velocity

$$G_t = \frac{\dot{m}}{a_t} = \frac{\dot{m}_{vo} + \dot{m}_{Lo}}{a_t} = 146.04 \text{ kg}/(\text{m}^2 \cdot \text{s})$$

Tube side velocity,

$$u_t = \frac{G_t}{\rho_{av}} = 1.14 \text{ m/s}$$

$$Re = \frac{d_i G_t}{\mu_L} = \frac{0.022098 \times 162.04}{0.00032} = 11,637$$

(It is a thumb rule to use liquid viscosity, instead of liquid-vapour mixture)

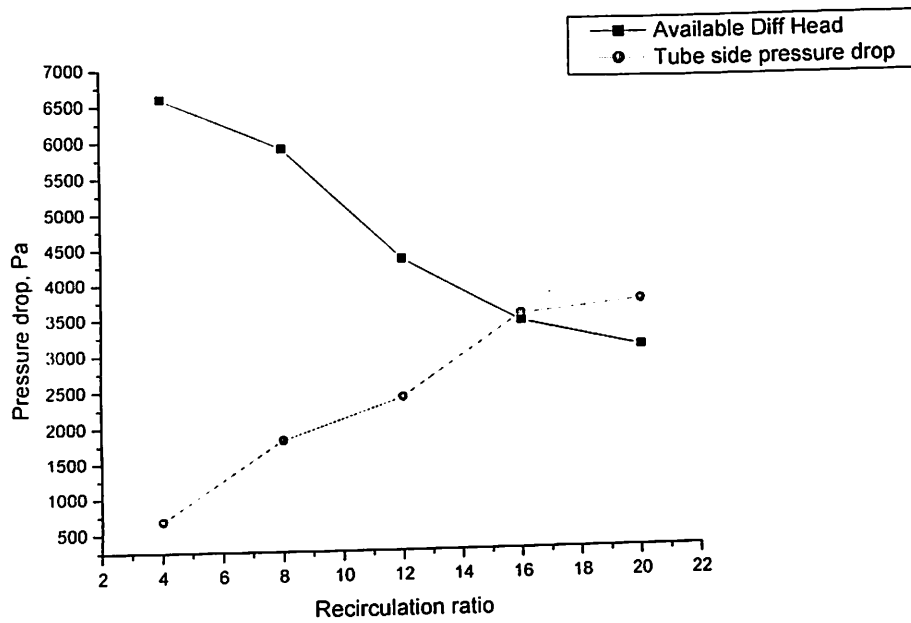
From fig.

$$J_f = 5 \times 10^{-3}$$

$$\Delta P_t = N_p \left(8 J_f \left(\frac{L}{d_i} \right) \left(\frac{\mu}{\mu_w} \right)^{-m} + 2.5 \right) \frac{\rho_{av} u_t^2}{2} = 678 \text{ Pa}$$

$$\Delta P_a > \Delta P_t$$

Trial	Recirculation ratio	ΔP_a , Pa	ΔP_t , Pa	Condition satisfied
1	4	6590	678	No
2	8	5892	1813	No
3	12	4458	2397	No
4	16	3487	3592	Yes



7.3 Pressure drop Vs Recirculation ratio

Heat transfer coefficients:

(a) Tube side heat transfer coefficient

$$Re = 33265$$

$$Pr = \frac{C_L \mu_L}{k_L} = 5.44$$

(Using liquid properties)

Properties of liquid mixture at 120°C:

Specific heat, $C_L = 3.4 \text{ kJ}/(\text{kg} \cdot ^\circ\text{C})$

Viscosity, $\mu_L = 0.32 \text{ cP}$

Thermal conductivity, $k_L = 0.2 \text{ W}/(\text{m} \cdot ^\circ\text{C})$

The convective boiling co-efficient is calculated using Chen's method.

As the heat flux is known ($37900 \text{ W/m}^2\text{C}$), only a rough estimate of the nucleate boiling is required.

Using Mostinski equation to estimate the nucleate boiling coefficient:

Critical Pressure = 8.056 bar

$$h_{nb} = 0.104(P_c)^{0.69}(q) \left[1.8 \left(\frac{P}{P_c} \right)^{0.17} + 4 \left(\frac{P}{P_c} \right)^{1.2} + 10 \left(\frac{P}{P_c} \right)^{10} \right]$$

$$h_{nb} = 1528.09 \text{ W/m}^2\text{.}^0\text{C}$$

Vapour Quality, $x = 0.06$

$$\mu_v = 0.0084 \text{ mN}$$

$$\frac{1}{X_{tt}} = \left(\frac{0.06}{1 - 0.06} \right)^{0.9} \times \left(\frac{786.1}{8.1} \right)^{0.5} \times \left(\frac{0.0084}{0.12} \right)^{0.5} = 0.6347$$

For $Nre = 33265$, $J_H = 0.0033$

$$Nu = J_H \cdot Nre \cdot Pr^{0.33} = 191.8$$

$$h_i = Nu \left(\frac{K_L}{D_i} \right) = 1738 \text{ W/m}^2\text{.}^0\text{C}$$

Convective boiling enhancement factor, $f_c = 2.9$ (From ref-29, Fig. 12.56)

$$Re_L \cdot f_c^{1.25} = 125888$$

Nucleate boiling suppression factor, $f_s = 0.21$

$$h_{cb} = f_c h_i + f_s h_{nb} = 3970.7 \text{ W/m}^2\text{.}^0\text{C}$$

This value has been calculated at outlet condition.

Assuming coefficient changes linerly for the inlet and outlet, then

$$\text{Average coefficient} = \frac{(\text{Inlet Coefficient of liquid (all liquid)} + \text{outlet coefficient (two phase)})}{2}$$

$$N_{re} \text{ at inlet} = 51560$$

$$J_H = 0.0032$$

$$Nu = 270.47$$

$$H_i = 2447.91 \text{ W/m}^2 \cdot ^\circ\text{C}$$

$$\text{Mean co-efficient} = 3208.95 \text{ W/m}^2 \cdot ^\circ\text{C}$$

$$\text{Shell side heat transfer coefficient } h'_o = 6000 \text{ W/(m}^2 \cdot ^\circ\text{C)}$$

(This value includes fouling resistance)

$$\frac{1}{U_o} = \frac{1}{h'_o} + \frac{d_o \ln\left(\frac{d_o}{d_i}\right)}{2k_w} + \frac{d_o}{d_i} \times \frac{1}{h_i} + \frac{d_o}{d_i} \times \frac{1}{h_{id}}$$

Thermal conductivity of tube material $k_w = 45 \text{ W/(m}^2 \cdot ^\circ\text{C)}$ (Mild steel tubes)

$$\frac{1}{U_o} = \frac{1}{6000} + \frac{0.0254 \times \ln\left(\frac{0.0254}{0.022098}\right)}{2 \times 45} + \frac{0.0254}{0.022098} \times \frac{1}{3208.95} + \frac{0.0254}{0.022098} \times \frac{1}{5000}$$

$$U_o = 1259.1 \text{ W/(m}^2 \cdot ^\circ\text{C)}$$

Heat transfer area required

$$A_{req} = \frac{Q}{U_o \Delta T_m} = \frac{36.96 \times 1000}{1002.2 \times 39.6} = 0.74 \text{ m}^2$$

$$\frac{A_{pro}}{A_{req}} = \frac{0.9335}{0.74} = 1.26$$

Heat transfer area required $A_{req} = 0.74 \text{ m}^2$

Heat transfer area provided $= \pi N d_o L = 0.9335 \text{ m}^2$

$$\% \text{ Excess heat transfer area} = \left(\frac{A_{pro}}{A_{req}} - 1 \right) \times 100 = \left(\frac{0.0335}{0.74} - 1 \right) \times 100 = 26.14 \%$$

Shell side pressure drop Δp_s :

$$\Delta p_s = 0.5 \times 8 \times J_f \left(\frac{D_s}{d_e} \right) \left(\frac{L}{B_s} \right) \frac{\rho u_s^2}{2}$$

Shell side Friction factor, J_f ,

To find this factor, first shell side flow area must be determined.

$$A_s = \frac{(P_t - d_o) D_s B_s}{P_t}$$

Let $B_s =$ Baffle spacing $= D_s =$ Shell inside diameter

$$B_s = D_s = 0.438 \text{ m}, d_o = 25.4 \text{ mm}, P_t = 31.75 \text{ mm}$$

$$A_s = \frac{(31.75 - 25.4) \times 0.203^2}{31.75} = 0.00825 \text{ m}^2$$

$$\text{Shell side mass velocity } G_s = \frac{\dot{m}_s}{A_s} = \frac{0.0238}{0.00825} = 2.89 \text{ kg}/(\text{m}^2 \cdot \text{s})$$

$$\text{Density of steam } \rho_v = \frac{1}{\text{specific volume}} = \frac{1}{0.46511} = 2.15 \text{ kg}/\text{m}^3$$

From Steam tables specific volume of saturated steam at 510 kPa pressure, $v_s = 0.46511 \text{ m}^3/\text{kg}$

$$u_s = \frac{G_s}{\rho_v} = \frac{2.88}{2.15} = 1.339 \text{ m/s}$$

Equivalent diameter,

$$d_e = \frac{1.1}{d_o} (P_t^2 - 0.907 d_o^2) = \frac{1.1}{0.0254} (0.03175^2 - 0.907 \times 0.0254^2) = 0.0183 \text{ m}$$

Shell side Reynolds number:

$$Re_s = \frac{d_e G_s}{\mu}$$

Steam viscosity at 120° and 510 kPa, $\mu_v = 18.5 \times 10^{-6} \text{ (N.s)/m}^2$

Ref: Table 3.302. Perry Handbook

$$Re_s = \frac{d_e G_s}{\mu} = 13675$$

$J_f = 4.78 \times 10^{-2}$ (For 25% baffle cut)

$$\Delta p_s = 0.5 \times 8 \times J_f \left(\frac{D_s}{d_e} \right) \left(\frac{L}{B_s} \right) \frac{\rho u_s^2}{2} = 37.73 \text{ Pa}$$

$\Delta p_s \ll 13.8 \text{ kPa}$

It is well within permissible limit.

8. SCALE UP OF HYDRAZINE HYDRATE PROCESS

Plant sizes are usually characterized by their production capacity or throughput. Define the *throughput scaleup factor* as

$$S_{throughput} = \frac{\text{mass flow through full - scale unit}}{\text{mass flow through pilot}} = \frac{(\rho Q)_{\text{full scale}}}{(\rho Q)_{\text{pilot scale}}} \dots \dots (8.1)$$

Assume that the pilot-scale and full-scale vessels operate with the same inlet density.

Then ρ cancels in Equation 1.55 and

$$S_{throughput} = \frac{(Q)_{\text{full scale}}}{(Q)_{\text{pilot scale}}} \dots \dots (8.2)$$

There is also an inventory scaleup factor defined as

$$S_{inventory} = \frac{\text{mass inventory in full - scale unit}}{\text{mass inventory in pilot - scale unit}} = \frac{(\hat{\rho}V)_{\text{full scale}}}{(\hat{\rho}V)_{\text{pilot scale}}} \dots \dots (8.3)$$

For successful scaleup we expect the volume-average density $\hat{\rho}$ to be the same in the large and small reactors. Thus, normally

$$S_{inventory} = S_{volume} = \frac{V_{\text{full scale}}}{V_{\text{pilot scale}}} \dots \dots (8.4)$$

8.1 Reactors:

The product from the scaledup reactor should be the same as the product from the pilot reactor.

The extent of reaction should be the same in the two reactors and thus the mean residence time \bar{t} should held constant upon scaleup.

The *mean residence time* \bar{t} is the average amount of time that a material spends in a reactor. The concept of mean residence time is most useful for flow systems. In a steady-state flow system, \bar{t} is equal to the mass inventory of fluid in the system divided by the mass flow rate through the system:

$$\bar{t} = \frac{\text{mass inventory}}{\text{mass throughput}} = \frac{\hat{\rho}V}{\hat{\rho}Q} \dots \dots (8.5)$$

According to the Equation 5, inventory and throughput are increased by the same factor when \bar{t} is held constant. Unless explicitly stated otherwise, it is understood that inventory, volume, and throughput all increase proportionately:

$$S_{throughput} = S_{inventory} = S_{volume} = S \dots \dots \dots (6)$$

Use of S without subscripts indicates that Equation 6 is satisfied. This is true for the great majority of reactor scaleups for both liquids and gases.

It is common practice to use *geometric similarity* in the scaleup of stirred tanks (but not tubular reactors). Geometric similarity means that the production-scale reactor will have the same shape as the pilot-scale reactor. All linear dimensions such as reactor diameter, impeller diameter, and liquid height will change by the same factor, $S^{1/3}$. Surface areas will scale as $S^{2/3}$.

The full correlation shows t_{mix} to be a complex function of the Reynolds number, the Froude number, the ratio of tank to impeller diameter, and the ratio of tank diameter to liquid level. However, to a reasonable first approximation for geometrically similar vessels operating at high Reynolds numbers,

$$(NI t_{mix})_{large} = \text{const} = (NI t_{mix})_{small} \dots \dots \dots (8.7)$$

where NI is the rotational velocity of the impeller. This means that scaleup with constant agitator speed will, to a reasonable approximation, give constant t_{mix} .

The rub is that the power requirements for the agitator will increase sharply in the larger vessel. To a reasonable first approximation for geometrically similar vessels operating at high Reynolds numbers,

$$\left(\frac{P_I}{\rho N_i^3 D_i^5} \right)_{large} = \left(\frac{P_I}{\rho N_i^3 D_i^5} \right)_{pilot} = P_0 \dots \dots \dots (8.8)$$

where P_I is the impeller power, D_I is the impeller diameter, and P_0 is a dimensionless group known as the *power number*. If N_I is held constant, the impeller power will increase as D_I^5 and as $S^{5/3}$ since DI scales as $S^{1/3}$ using geometric similarity. A factor of 10 increase in the linear dimensions gives $S = 1000$ and allows a factor of 1000 increase in throughput. Unfortunately, it requires a factor of 100,000 increase in agitator power! The horsepower per unit volume must increase by a factor of 100 to maintain a constant t_{mix} . Most scaleups are done with approximately constant power per unit volume, which causes N' to decrease and t_{mix} to increase upon scaleup.

8.2 Effect on RTD:

The pilot reactor is a CSTR. The large reactor will be geometrically similar to the small one, and the scaleup will be done at constant power per unit volume. This form of scaleup exploits the fact that small vessels typically use impeller speeds that are faster than necessary to achieve a close approach to the exponential distribution. Scaleup will eventually cause problems because the ratio of circulation to throughput scales with the impeller speed, and this will decrease when scaling at constant power per unit volume. Correlations exist for the pumping capacity of common impellers, so that the ratio of circulation to throughput can be calculated. If it is maintained at a reasonable value, say 8 : 1, the RTD will remain close to exponential. Better, the impeller speed can be decreased in the small unit to anticipate what will be feasible in the large unit. Decrease it by a factor of $S^{2/9}$, where S is the intended scaleup factor for throughput as shown in the following table. Scaling with constant power per unit volume will maintain the same Kolmogorov eddy size, so that micromixing should not become a problem.

8.3 Scale up calculations for CSTR*:

Plant sizes are usually characterized by their production capacity or throughput. Define the *throughput scaleup factor* as

$$S_{\text{throughput}} = \frac{\text{Mass flow through pilot scale unit}}{\text{Mass flow through bench scale unit}} = (100 / 7.47) = 13.38$$

	General scaling factor	Scaling factor for constant power per unit volume	Numerical scaling factor (S=13.38)	At bench scale	At pilot scale from scale up factor	Calculated for pilot plant
Vessel diameter	$S^{1/3}$	$S^{1/3}$	2.37	300 mm	711mm	800 mm
Impeller diameter	$S^{1/3}$	$S^{1/3}$	2.37	150 mm	355.5mm	266 mm
Vessel volume	S	S	13.38	25 lit	334.5 lit	630 lit
Through put	S	S	13.38	1.723 kg/hr	100 kg/hr	100 kg/hr
Residence time	1	1	1	8 hrs	8 hrs	8 hrs
Reynolds number	$S_N S^{1/3}$	$S^{4/9}$	3.1669	117187	371120	246335
Agitator speed	S_N	$S^{-2/9}$	0.5619	600 rpm	337.14 rpm	400 rpm
Power	$S^3_N S^{5/3}$	S	13.38	0.381 hp	5.09 hp	3.022 hp
Power per volume	$S^3_N S^{2/3}$	1	1	0.01524	0.01524	0.0177

The similarities followed in the scale-up to 3-reactor system:

i) Geometric Similarity:

Straight height of the tank / Tank diameter

Liquid Level Height / Tank Diameter

Type of impeller

Impeller Diameter / Tank Diameter

Baffles

ii) Mixing Similarity:

Tip Speed

Power required per unit volume of Reaction mixture

Volumetric flowrate per velocity head

iii) Kinetic Similarity:

Reaction time

Reaction temperature

Reaction pressure

Reactants flow rate

iv) Ammonia Sparger Similarity:

Sparger ring diameter

Orifice diameter

Orifice Reynolds number

v) Thermal Similarity:

Heat transfer rate

The respective values for the above similarities for the three different scales are given below:

i) Geometrical similarities:

	Semi batch Reactor	Continuous Reactor	Pilot plant 3-Reacto system
Impeller Dia / Tank dia (D_i / D_T)	0.621	0.500	0.333
Liquid depth / Tank dia (H_L / D_T)	1.580	0.493	0.570
Overflow height / Tank dia	-- --	0.691	0.788
Type of Impeller	Propeller	Flat blade	Flat blade

ii) Mixing similarities:

	Semi batch Reactor	Continuous Reactor	Pilot plant 3-Reacto system
Tip Speed (ft/m) ($\Pi Da N$)	630	927	1100
RPM	1200	600	400
Reynolds Number ($Da^2 N \rho / \mu$)	2.7×10^3	1.17×10^5	2.46×10^5
Power (hp) ($PN \rho N^3 Da^5$) ($PN = 6.1$ for $NRe > 10^4$)	0.0212	0.582	3.022

iii) Sparger similarities:

	Semi batch Reactor	Continuous Reactor	Pilot plant 3-Reactor system
Orifice diameter	0.5 mm	0.5 mm	1.0 mm
Number of orifices	3	6	46 (1st reactor) 23 (2nd & 3rd)
Orifice NRe	7,682	15,364	2.35×10^5

9. CONCLUSIONS:

The following conclusions can be drawn from this present work:

Experiments were done to measure the transport properties of the intermediate compounds.

The following equipments have been designed based on IS-4179:

- ✓ Storage Tanks – 54
- ✓ Stirred vessels – 13
- ✓ Packed towers– 5
- ✓ Thermosyphon Reboiler – 5
- ✓ Horizontal condenser – 5
- ✓ Crystallizers – 6
- ✓ Line sizing, Orifice sizing of Section-100 and Section-200
- ✓ Pump selection of the all four section
- ✓ Design were done on hand calculation and transformed into spreadsheet.
- ✓ Specification sheets have been submitted to the concerned department for tendering.
- ✓ Scale up of reactor was done based on geometric similitude.

10. RECOMMENDATIONS:

Hydrazine hydrate process has two reactions, viz first step oxidation of ammonia with hydrogen peroxide as Methyl Ethyl Ketone catalyst and Acetamide being co-catalyst. It has been experimentally observed that acetamide recovery is 74%, alternative catalyst can be sought in order to overcome this problem. Use of formamide is recommended as it reduces the reaction time to 6 hours from 8 hours also its consumption is just 1 mole whereas acetamide is used at ratio of 2.5 to peroxide.

In the second step reaction, i.e. hydrolysis of methyl ethyl ketazine which is an autocatalytic reaction, the reaction can be enhanced by using DK10 weak acid type ion-exchange resin as catalyst.

BIBLIOGRAPHY:

1. D. Altman and B. Adelman, *J. Am. Chem. Soc.* 74, 3742 (1952).
2. E. W. Schmidt. *Hydrazine and its Derivatives*, 2nd ed., John Wiley & Sons, Inc., New York, 2001, pp. 191, 199, 299.
3. L. Pederson and K. Morokuma, *J. Chem. Phys.* 46, 3941 (1967).
4. G. H. Hudson, R. C. H. Spencer, and J. T. P. Stern, *Mellor's Comprehensive Treatise on Inorganic and Theoretical Chemistry*, Vol. 8, Suppl. 2, John Wiley & Sons, Inc., New York, 1967, pp. 69-113.
5. K. Jones, in J. C. Bailor, ed., *Comprehensive Inorganic Chemistry*, Vol. 2, Pergamon Press, Ltd., Oxford, U.K., 1973, p. 255.
6. M. W. Chase, ed., *NIST JANAF Thermochemical Tables*, JPCRD 1998, Monograph 9.
7. Y. Morino and co-workers, *Bull. Chem. Soc. Jpn.* 33, 46 (1960).
8. *Liquid Propellants Manual*, contract NOW 62-0604-c, Chemical Propulsion Information Agency, Johns Hopkins University, Baltimore, Md., 1961, Unit 2 (AH), Unit 5 (UDMH) and Unit 11 (MMH); *Hazards of Chemical Rockets and Propellants Handbook*, Vol. 3, CPIA/194, publ. no. AD 870259, National Technical Information Service, U.S. Dept. of Commerce, Washington, D.C., May 1972, Unit 9 (AH and MMH), Unit 10 (UDMH)
9. H. O. House, *Modern Synthetic Reactions*, 2nd ed., W. A. Benjamin, Inc., Menlo Park, Calif., 1972, pp. 228-256.
10. Ref. 2, exhaustive bibliography.
11. F. E. Scott, J. J. Burns, and B. Lewis, *Explosive Properties of Hydrazine*, Report of Investigations 4460, U.S. Dept. of the Interior, Bureau of Mines, Pittsburgh, Pa., May 1949.
12. W. M. Latimer, *The Oxidation States of the Elements and Their Potentials in Aqueous Solutions*, 2nd ed., Prentice Hall, Inc., Englewood Cliffs, N.J., 1952, p. 99.
13. L. F. Audrieth and B. A. Ogg, *The Chemistry of Hydrazine*, John Wiley & Sons, Inc., New York, 1951, pp. 86-94.

14. M. L. Davis, V. V. Vesselovsky, and H. L. Johnston, Kinetics, Thermodynamics, Physico-Chemical Properties and Manufacture of Hydrazine, Ohio State University, Wright-Patterson Air Force Base, Dayton, Ohio, Mar. 15, 1952.
15. *Ber. Ger. Patent 19830, 1907.*
16. O. M. Arnold. G. Pointe, R. M. Jamison, *U.S. Patent 2715564, 1955.*
17. Nikitha P., Saibabu K.B.S., Techno-Economic Analysis of Hydrazine Hydrate Technologies, *Chem. Eng. Technol.* 2010, 33, No. 9, 1543–1551.
18. Raminder Kaur, Ramakrishna Machiraju and K. D. P. Nigam, Kinetics Studies of Ketazine Formation: Effect of Temperature and Catalyst Concentration, *Can. J. Chem. Eng.* 86:99–104, 2008.
19. Olin Corp. Product Bulletin 731-009R3, Hydrazine Storage and Handling of Aqueous Solutions, 1991. For propellant hydrazines (AH, MMH, UDMH), see Air Force Systems Command Design Handbook, 1–6, System Safety, 5th ed., Rev. 2, Wright Patterson Air Force Base, Dayton, Ohio, Apr. 13, 1990.
20. G. N. Kumar and co-workers, *Curr. Sci.* 57(9), 1069 (1988).
21. C. O. Arah and co-workers, *Int. SAMPE Symp. Exhib.* 36(2), 1545 (1991).
22. J. M. Bellerby, *J. Hazard. Mater.* 7(3), 187 (1983).

APPENDIX 1: About IICT

Council of Scientific and Industrial Research is largest publicly funded organisation in the world having 39 National Laboratories in India and Indian Institute of Chemical Technology is one of the premier constituent laboratories of CSIR. Laboratories of CSIR Conduct Research in the areas of Physical, Chemical, Biological and Information Sciences.

Indian Institute of Chemical Technology, Hyderabad is a premier R&D Institute in India. The Institute had its origin as the Central Laboratories for Scientific & Industrial Research (CLSIR), established in 1944 by the then Government of Hyderabad State. After integration of Hyderabad State with the Indian Union, the laboratory expanded with its growing activities. The main building was formally opened by Pundit Jawaharlal Nehru, the then Prime Minister of India on January 2, 1954. In 1956, the Central Laboratories came under the aegis of the Council of Scientific & Industrial Research (CSIR), New Delhi and was renamed Regional Research Laboratory, Hyderabad (RRL-H). The RRL-H was rechristened as the Indian Institute of Chemical Technology (IICT), Hyderabad in 1989, recognizing the multidisciplinary activities and the expertise developed by the Institute in the field of chemical technology. Major areas of research at IICT are: Natural Products Chemistry, Agrochemicals, Drugs & Intermediates, Specialty and Fine Chemicals, Fluoroorganics, Inorganic & Physical Chemistry (Catalysis & Material Science), Lipid Sciences & Technology, Coal, Gas & Energy, Chemical Engineering and Design & Engineering.

IICT's basic objectives have always been to carry out research in the chemical sciences leading to innovative processes for a variety of products necessary for human welfare such as food, health and energy and the conduct of R&D work is fully geared to meet the requirements of technology development, transfer and commercialisation. Process development work, particularly for bulk chemicals is carried out at appropriate pilot plant scale to collect techno-economic and design data. With the help of excellent design & engineering expertise available, the Institute has been providing engineering designs for commercial plants with standard commercial guarantees. More than 150 technologies developed by IICT are now in commercial production. Its commitment to industry is reflected by way of several sponsored and consultancy projects received from the industry, year after year. The external cash flow of IICT for the year 2003-04 is around Rs.16.5 crores. With over 450 highly professional and dedicated scientists and technical officers/technicians, excellent laboratory and instrument facilities for research in chemical sciences and technology and allied sciences, IICT

APPENDIX 2: Spreadsheets

1. Line Sizing:

INDIAN INSTITUTE OF CHEMICAL TECHNOLOGY			
Prepared by: Ibrahim Khan		Column Tag No.:	T-602
Checked by: M Pradeep Kumar, M Naveen		Job No.:	170
Date:		Client:	GACL
		Project:	Hydrazine Hydrate

LIQUID LINE SIZING INPUT DATA			RESULTS			
Unit System to be Used	METRIC		Line No	0	Pipe Schedule	40
Pipe Material	Commercial Steel		Pipe - O.D./I.D./Wall Thickness			1.65 / 1.35 / 0.14 inches
Line No		Pipe Schedule	40			
Pressure	P	1.6	PARAMETERS		METRIC	FPS
Temperature	T	158	Pressure	P	1.6	22.7573
Mass Flow Rate	M	120	Temperature	T	158.00	312.8
Density	ρ_L	830	Flow Rate	M	120	264.555
Over Design Margin		5	Nominal Diameter	D	35	1.4
Viscosity	μ_L	1.13	Velocity	V	0.044	0.143
Max. Velocity	V_{max}	4	Pressure Drop	ΔP	13.0922	5.6752
Allowable Pressure Drop (per km of pipeline)	ΔP_{max}	30				

** Appropriate Schedule to be selected to avoid abnormal results

Mandatory Inputs

2. Packed Tower Sizing:

INDIAN INSTITUTE OF CHEMICAL TECHNOLOGY			
Prepared by: Ibrahim Khan		Column Tag No.:	T-602
Checked by: M Pradeep Kumar, M Naveen		Job No.:	170
Date:		Client:	GACL
		Project:	Hydrazine Hydrate

Input Data

Packing type =

Packing size =

Gas/Vapour Properties

Gas/Vapour flow = kg/h

= kg/s

Gas/Vap. Density = kg/m³

Liquid Properties

Liquid flow rate, L = kg/h

= kg/s

Liquid Density, ρ_L = kg/m³

Liquid Viscosity, μ = Ns/m²

Packing factor, F_p = m⁻¹

Conversion: C_g = Ns/m²

Calculation

Liq.-vap. Flow fact = $(L/V) \sqrt{(\sigma_v/\sigma_l)}$

=

Design for an initial pressure drop of 20mm H₂O/m packing
 From K_f vs $F_{L,0}$ [Click here for Graph](#)

K_f = 0.80

K_f at flooding = 1.10

Trial $F_{L, flooding}$ = $\sqrt{(K_f / K_f \text{ at flooding})} \times 100$
 = 85.2803

Gas mass flow rate = $\left[\frac{K_f \cdot \sigma_1 \cdot \sigma_2 \dots \sigma_n}{13.1 F_{L, flooding} \cdot \sigma_1 \cdot \sigma_2 \dots \sigma_n} \right]^{(1/2)}$
 = 1.3258 kg/m²s

Trial column area = V / V_L
 (Trial A_1) = 0.0003 m²

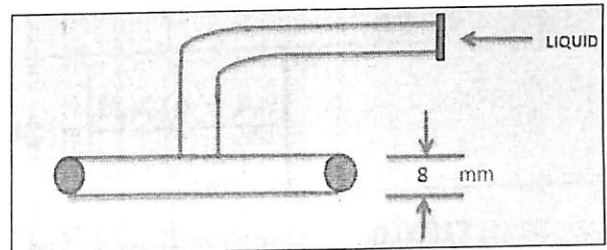
Trial column dia. = 0.0202 m $D = \sqrt{(4/p_i) \times \text{Trial } A_1}$

Round off D to nearest standard size
 Therefore, D = 0.100 m

Column C/S area, A_c = 0.0078 m² $A_c = (p_i/4) \times D^2$

Column Internals Design-Liquid Distributor

Quantity	Value	Units
Type	Pipe Type	
No. of Distribution Points	0.404845361	Nos
Round off to standard	4	Nos
Liq-Velocity through Pipe	2	m/s
Flow rate of Liquid	1.06936E-06	cu.m/sec
Inside diameter of pipe	0.000825299	m
	0.825298761	mm
Standard Size	8	mm
Liq-Velocity through Hole	3	m/s
Diameter of the Hole	0.000336927	m
	0.336926808	mm
Standard Size	3	mm



Column Internals Design-Packing Support

Assume

Quantity	Value	Units
Type	Gas Injection	Cap Type
Allowance for Flange	15	mm
OD of Packing Support	115	mm
Diameter of the riser	0.016666667	m
	16.66666667	mm
No. of Risers	9	Nos
Distance b/w two slots	5	mm
No. of Slots	5.233333333	Nos
Rounded off	6	Nos
% Void Space for packing	92	%
Height of the Slot in riser >=	26.74814815	mm
Rounded off	27	mm

Since it sandwiched

Selection of packing (T-304):

Type	1 inch pall ring
Packing factor, F_p (m^{-1})	170
Surface area, a (m^2/m^3)	194
Pressure drop, mm H ₂ O/ m of packing	50 (40 – 80 for distillation)

	RECTIFYING SECTION	STRIPPING SECTION
F_{LV}	0.0394	0.0853
K_4	1.75	1.8
$K_{4 \text{ Flooding}}$	5	3.9
%flooding $= \sqrt{\frac{K_4}{K_{4, \text{flooding}}}}$	59.16	67.94
Viscosity of liquid* from Souder equation	$\log(\log(10\mu)) = \frac{1}{M} \rho \times 10^{-3} - 2.9$	
Methanol, mN/m ²	0.308	0.277
Water, mN/m ²	0.3028	0.2755
Mixture, mN/m ²	0.308	0.2755
Gas flow-rate	$V_w^* = \left[\frac{K_4 \rho_v (\rho_l - \rho_v)}{13.1 F_p \left(\frac{\mu_L}{\rho_l} \right)^{0.1}} \right]^{1/2}$	
Gas flow-rate, V_w^* , kg/m ² sec	0.003	0.00317
V , kg/sec	0.0474	0.0474
Column area, m ²	0.06334	0.0668
Diameter, m	0.2838	0.2918
Onda's Method	$\frac{a_w}{a} = 1 - \exp \left[-1.45 \left(\frac{\sigma_c}{\sigma_l} \right)^{0.75} \left(\frac{L_w^*}{a \mu_l} \right)^{0.1} \left(\frac{L_w^{*2} a}{\rho_l g} \right)^{-0.05} \left(\frac{L_w^{*2}}{\rho_l \sigma_l a} \right)^{0.2} \right]$	
	$k_L \left(\frac{\rho_l}{\mu_l g} \right)^{1/3} = 0.0051 \left(\frac{L_w^*}{a_w \mu_l} \right)^{2/3} \left(\frac{\mu_l}{D_l \rho_l} \right)^{-1/2} (ad_p)^{-2.0}$	
	$\frac{k_G RT}{a D_v} = k_5 \left(\frac{V_w^*}{a \mu_v} \right)^{0.7} \left(\frac{\mu_v}{D_v \rho_v} \right)^{1/3} (ad_p)^{-2.0}$	
σ_c ss, N/m	61×10^3	61×10^3
σ_c , N/m	12.387×10^3	12.207×10^3
L_w^* , kg/m ² .sec	1.866	4.025
a , m ² /m ³	194	194
a_w , m ² /m ³	176.434	191.14

Estimation of Diffusivity for vapour and liquid		
Fuller eq.	$D_v = \frac{1.013 \times 10^{-17} T^{1.75} \left(\frac{1}{M_a} + \frac{1}{M_b} \right)^{1/2}}{P[(\sum v_i)a(\sum v_i)b]}$	
Wilke and Chang eq.	$D_L = \frac{1.173 \times 10^{-13} T(\phi M)^{0.5}}{\mu_m^{0.6}}$	
D_v , m ² /sec	4.446×10 ⁻⁵	4.707×10 ⁻⁵
ϕ	1	1
μ_L , Ns/m ²	0.308×10 ⁻³	0.2755×10 ⁻³
V_m , m ³ /Kmol	0.096	0.118
D_L , m ² /sec	4.966×10 ⁻⁹	5.501×10 ⁻⁹
μ_v , Ns/m ²	0.1003×10 ⁻³	0.1003×10 ⁻³
Mass transfer coefficients		
Liquid film, k_L , m/sec	9.928×10 ⁻⁴	3.8659×10 ⁻⁴
Gas film, k_G , m/sec	3.163×10 ⁻⁵	8.408×10 ⁻⁴
Film Transfer Unit Heights	$H_G = \frac{G_m}{K_G a_w P}$	$H_L = \frac{L_m}{K_L a_w C_t}$
G_m , Kmol/m ² .sec	0.0363	0.033
L_m , Kmol/m ² .sec	0.024	0.044
C_t , kmol/m ³	10.34	8.603
H_G , m	5.8364	33.243
H_L , m	0.134	0.069

3. Horizontal Condenser:

INDIAN INSTITUTE OF CHEMICAL TECHNOLOGY			
Design of Horizontal Condenser			
Prepared by:	Ibrahim Khan	Condenser Tag No: E-404	
Checked by:	M Pradeep Kumar, Naveen Kumar	Job No: 170000	
Date:		Client: GACL	
		Project: Hydrazine Hydrate	
INPUTS			
Process Fluid	Methane	Cooling Medium	Water
Flow rate	158 kg/hr	Flow rate	15489238 kg/s
Cp	5.2 kJ/kg	Cp	4.1868 kJ/kg
Density	796.1 kg/cu.m	Density	992.9 kg/cu.m
Viscosity	0.00011 kg/m.s	Viscosity	0.00072 kg/m.s
K	0.02 W/m degC	K	0.6228 W/m degC
Latent Heat of Vap	1100.56 kJ/kg	Latent Heat of Vap	2244 kJ/kg
Condensation	85 deg C	TD inlet	32 deg C
Subcooling	40 deg C	TD outlet	40 deg C
Liquid Prop at	57.3889-87.5 deg C	TD intermediate	31.8411561 deg C
Viscosity	0.00052 kg/m.s	R cond	863 mmHg
Density	805 kg/cu.m	K 55-302	16.3 W/m degC
K	0.2 W/m degC		
M.Wt	32.0		

Condenser Geometry Input	
OD	19.05 mm
Length	1 m
ID	15.748 mm
K1	0.319
n1	2.142 From table 1
Type	Fixed tube sheet
Shell Clearance	25 mm

4. Orifice Sizing:

INDIAN INSTITUTE OF CHEMICAL TECHNOLOGY				
Design of Orifice Meter Liquid				
Prepared by:	Ibrahim Khan	Orifice Tag No: O-101		
Checked by:	M Pradeep Kumar, Naveen Kumar	Job No: 170000		
Date:		Client: GACL		
		Project: Hydrazine Hydrate		
INPUTS		RESULTS		
Name of the Fluid	Water	Quantity	Value	Units
Flow rate	100000 kg/hr	Orifice Diameter	77	mm
Pipe ID	154 mm	Type	Circular	
SCH	40	Edges	Square	
Temperature, T	32 deg C	Radius Taps	Yes	
Density at T	995.026 kg/cu.m	Coefficient of Discharge	0.604166	
Viscosity at T	0.765 cP	Pressure Drop	4.681642	m WC
Manometric Fluid	Mercury	Manometer Reading	373.864	mm HG
Manometric Fluid Dens	13516.7 kg/cu.m	DP Transmitter	5507.814	mm WC

APPENDIX 3: Properties estimation using Aspen Plus

1. Hydrogen Peroxide:

Properties of Hydrogen peroxide is measured in the operating range of 25°C to 70°C

1.1 Density:

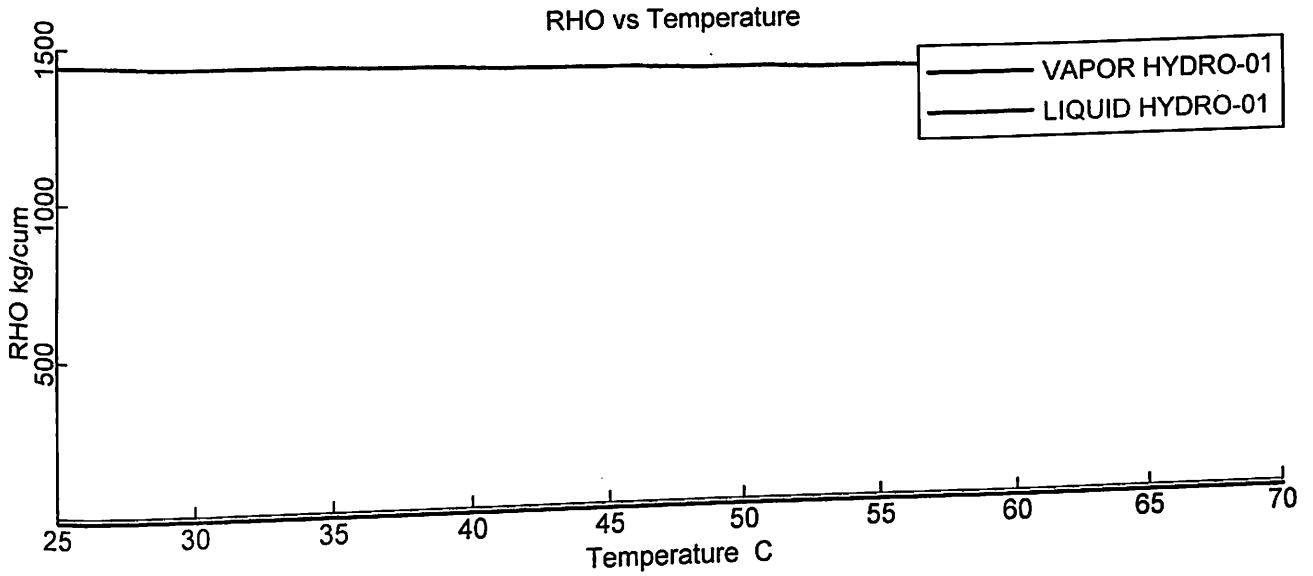


Fig. 3.1 Density of H₂O₂ Vs Temperature

1.2 Viscosity:

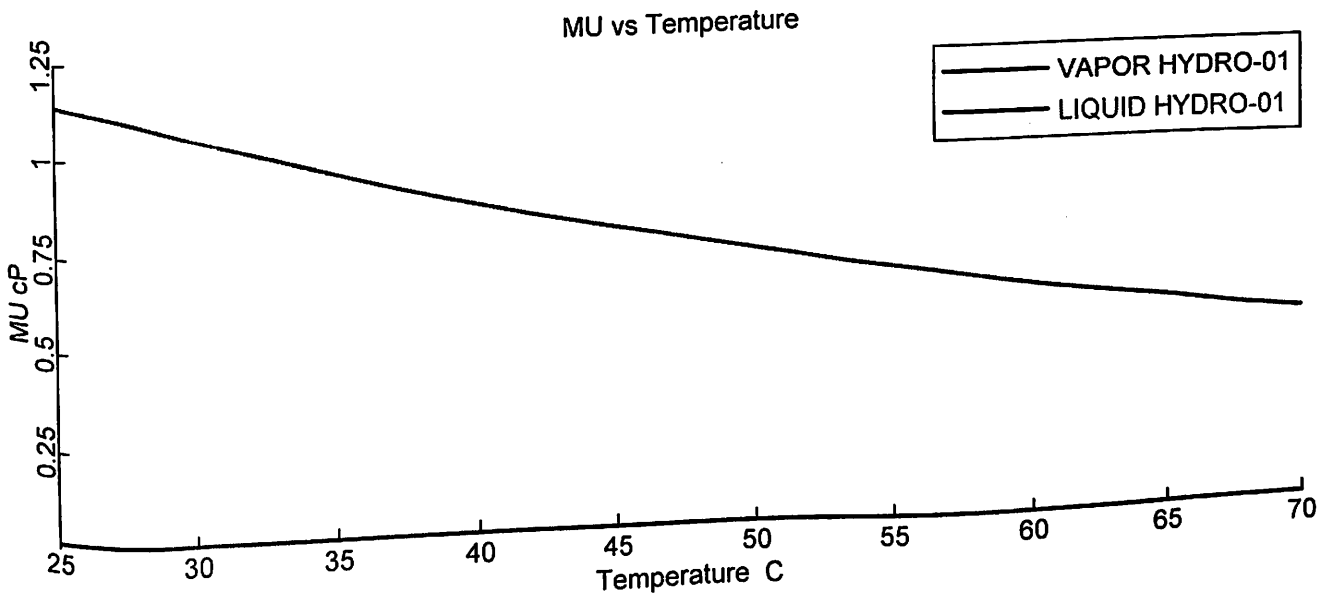


Fig. 3.2 Viscosity of H₂O₂ Vs Temperature

1.3 Specific Heat:

CP vs Temperature

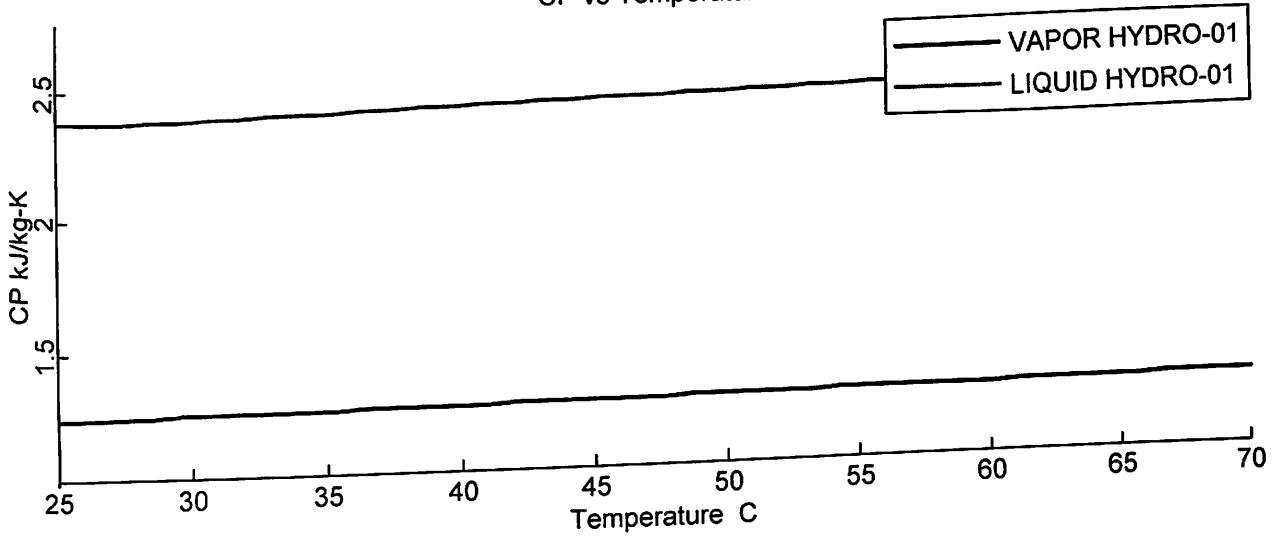


Fig. 3.3 Specific Heat of H2O2 Vs Temperature

1.4 Thermal Conductivity:

K vs Temperature

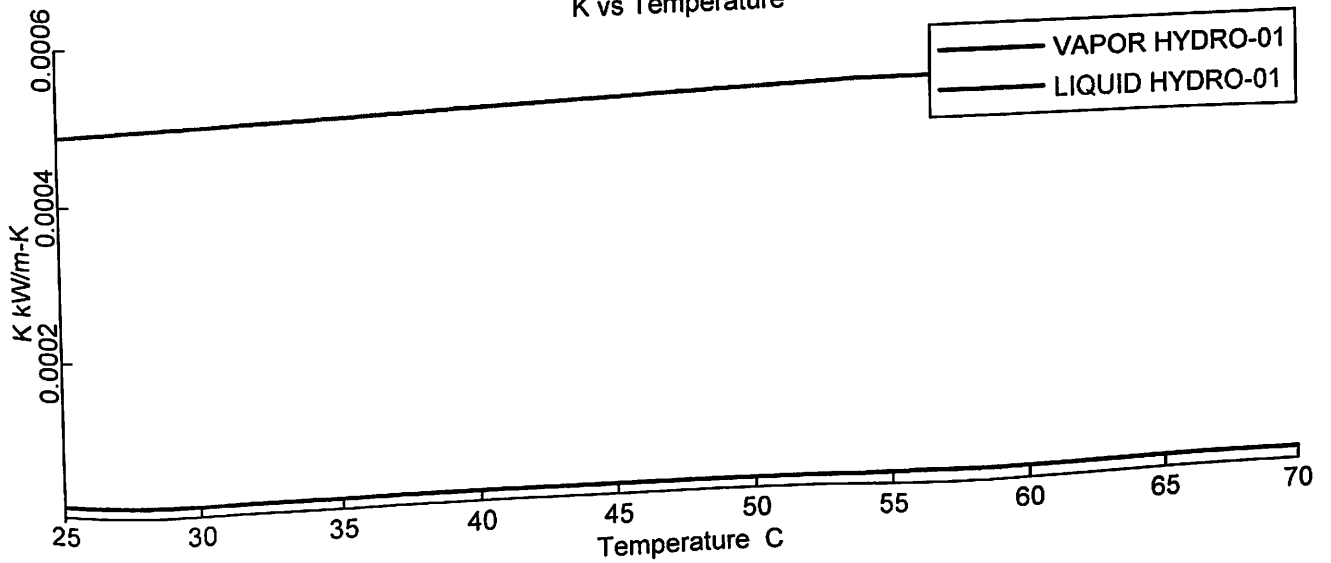


Fig. 3.4 Thermal Conductivity of H2O2 Vs Temperature

2. Methyl Ethyl Ketone:

2.1 Density:

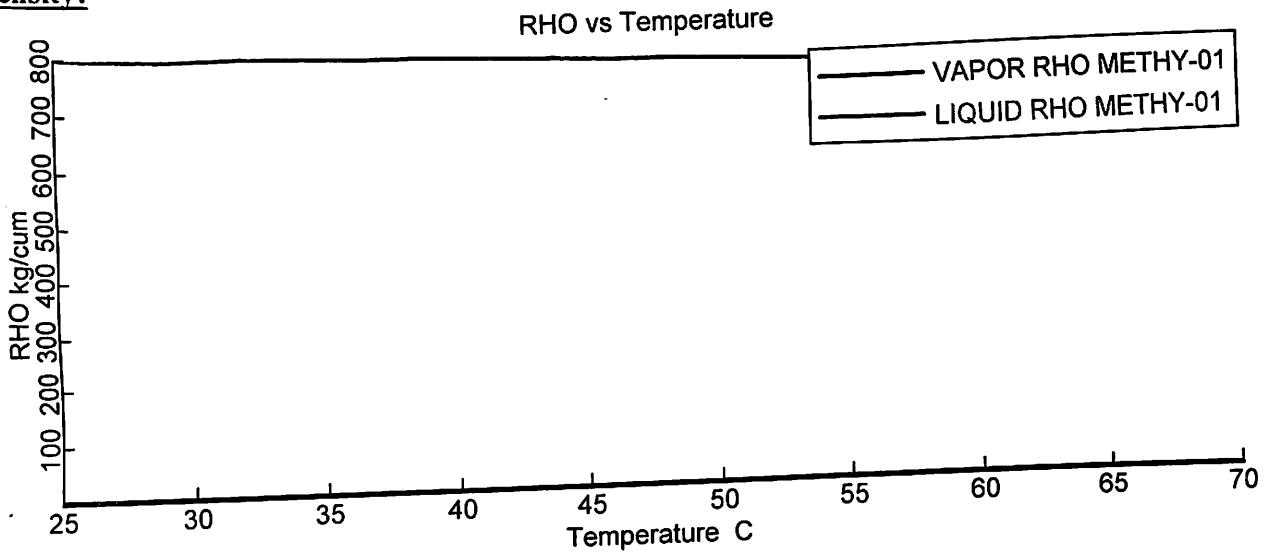


Fig. 3.5 Density of MEK Vs Temperature

2.2 Viscosity:

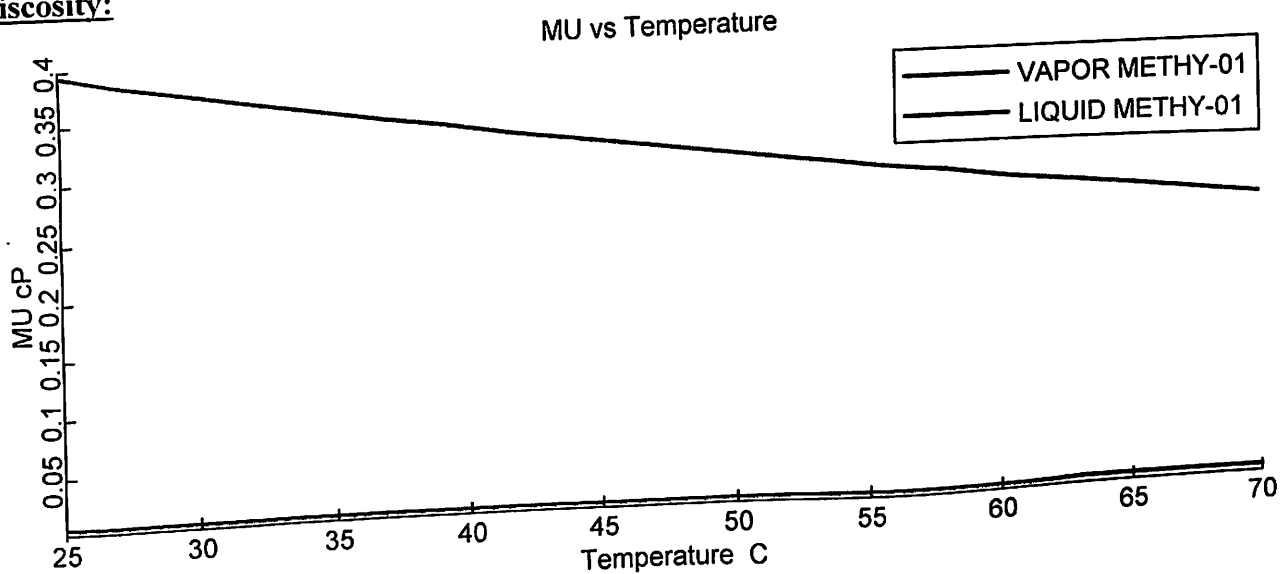


Fig. 3.6 Viscosity of MEK Vs Temperature

2.3 Heat Capacity:

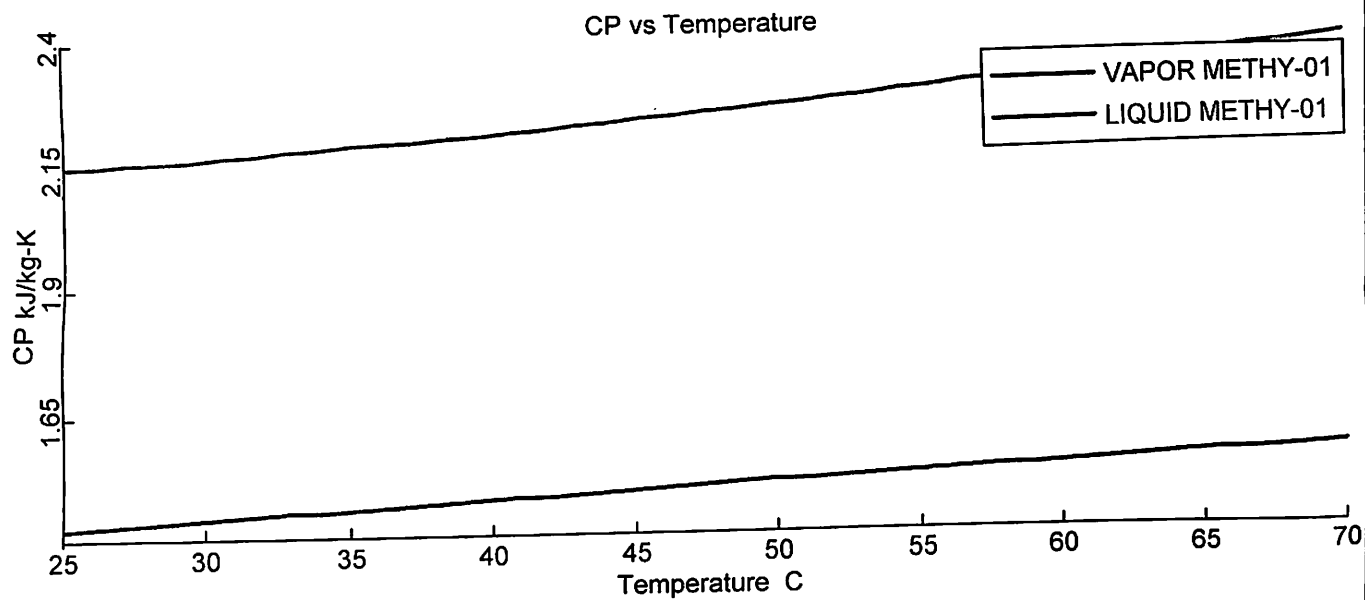


Fig. 3.7 Specific Heat of MEK Vs Temperature

2.4 Thermal Conductivity:

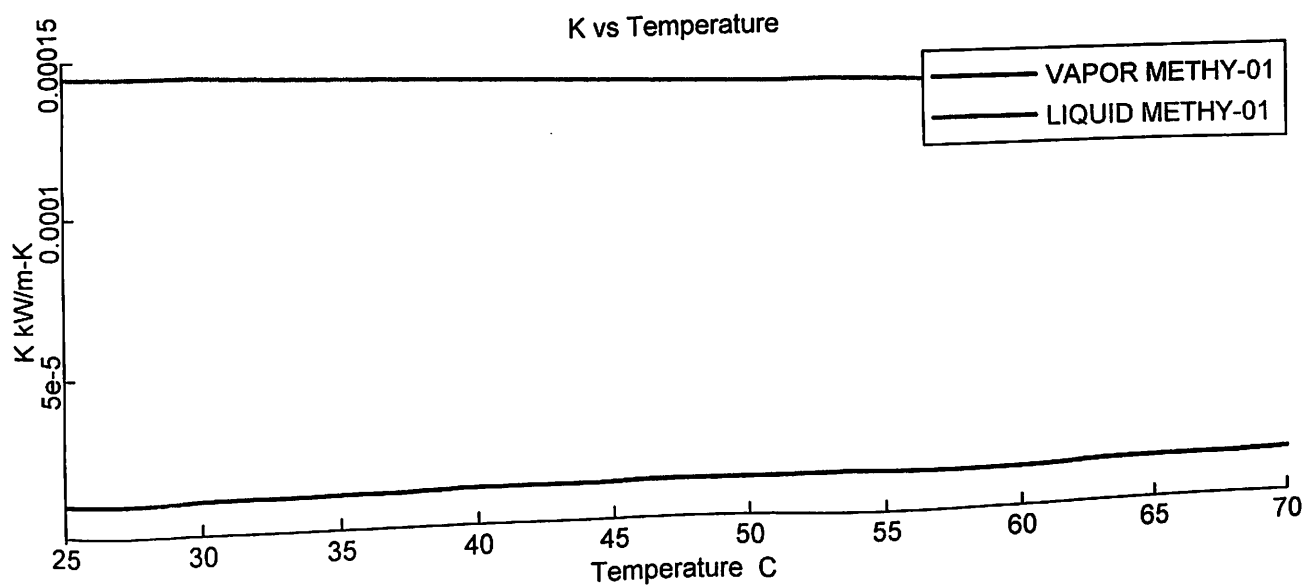


Fig. 3.8 Thermal Conductivity of MEK Vs Temperature

3. Acetamide:

3.1 Density:

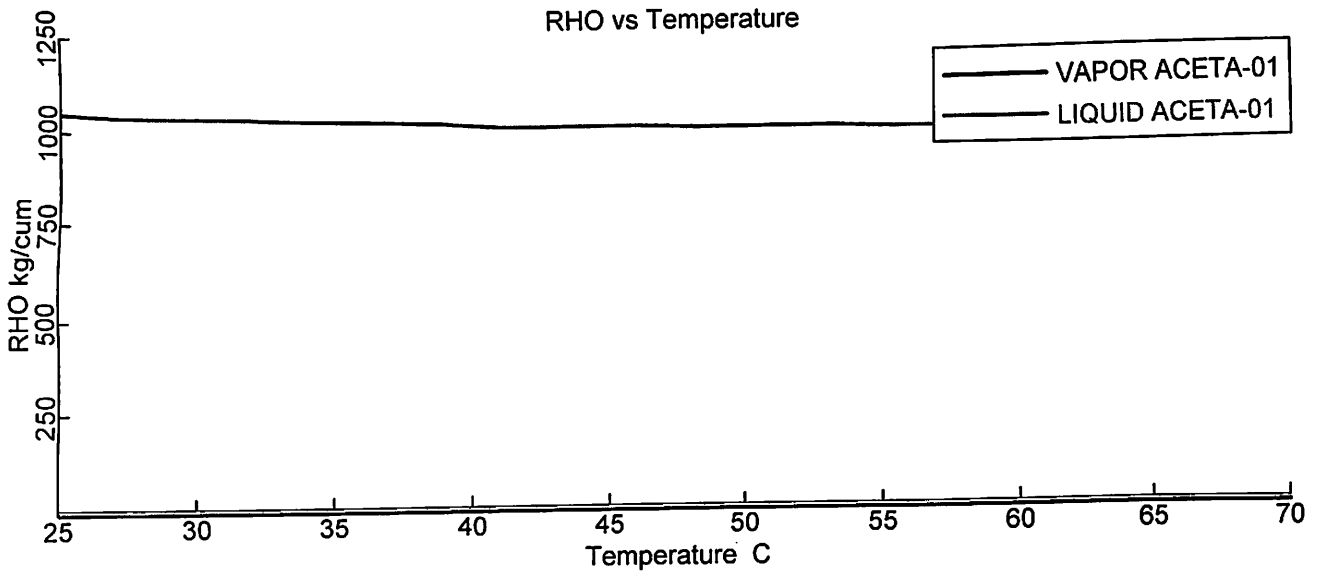


Fig.3.9 Density of Acetamide Vs Temperature

3.2 Viscosity:

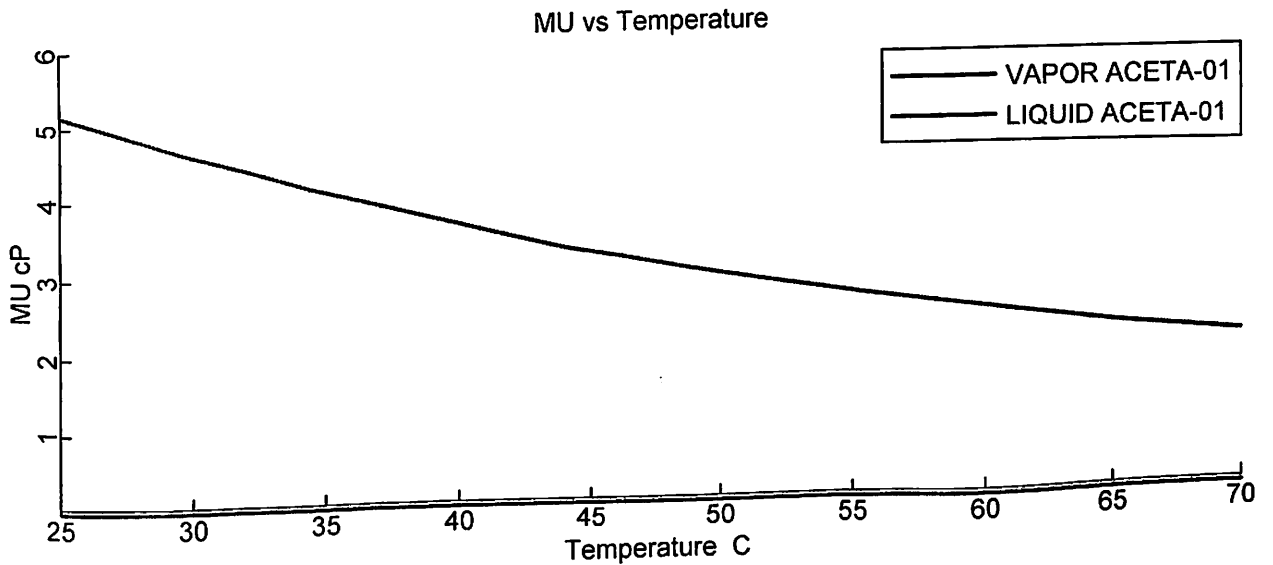


Fig.3.10 Viscosity of Acetamide Vs Temperature

3.3 Specific Heat:

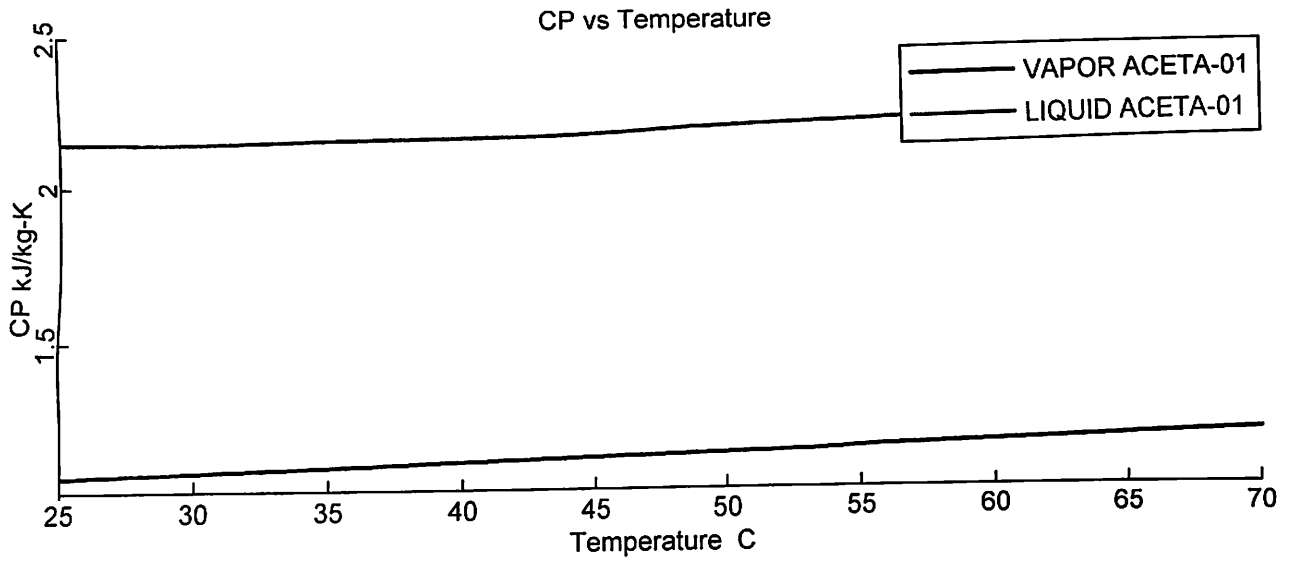


Fig.3.11 Specific Heat of Acetamide Vs Temperature

3.4 Thermal Conductivity:

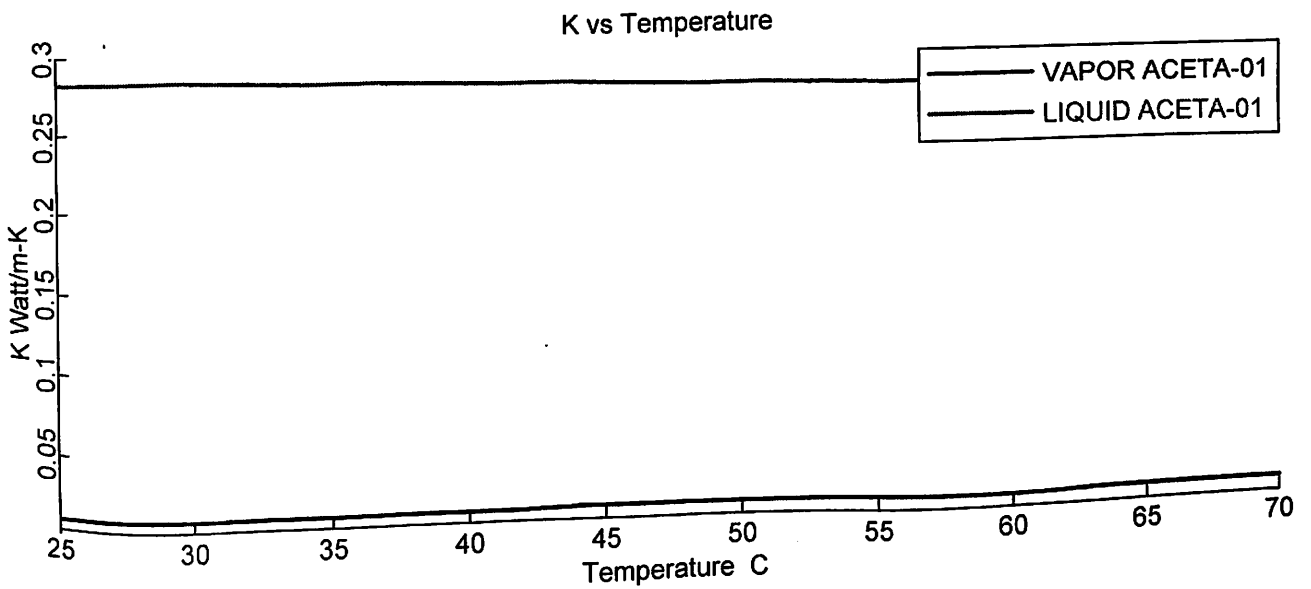


Fig.3.12 Thermal Conductivity of Acetamide Vs Temperature

4. Water:

4.1 Density:

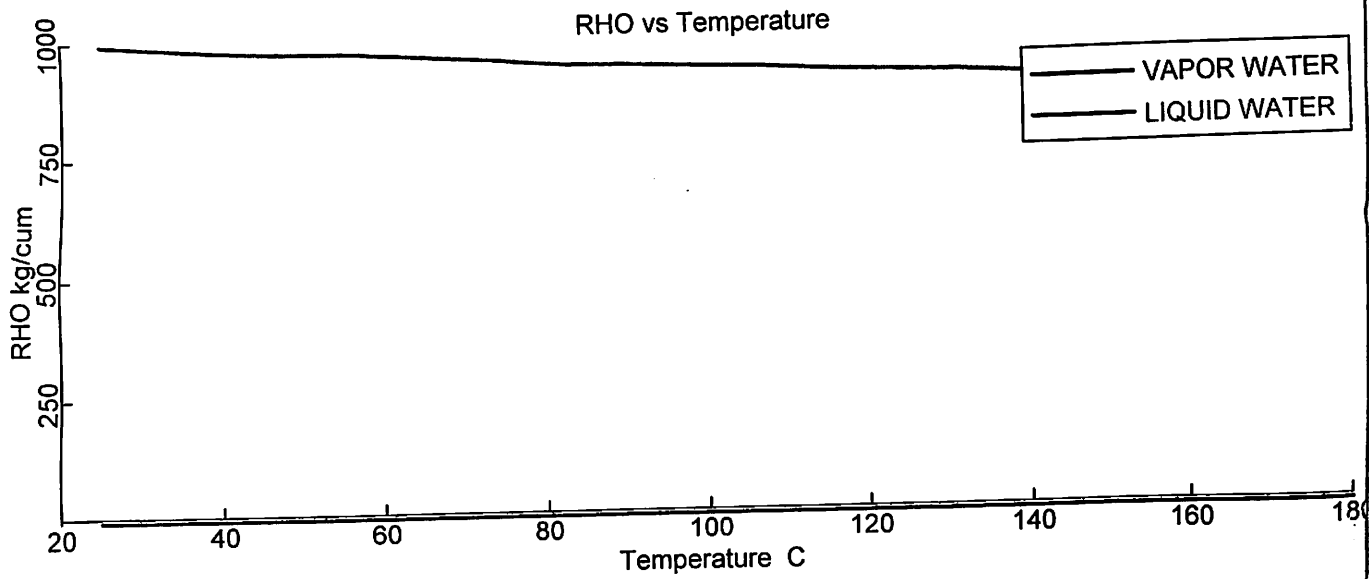


Fig.3.13 Density of Water Vs Temperature

4.2 Viscosity:

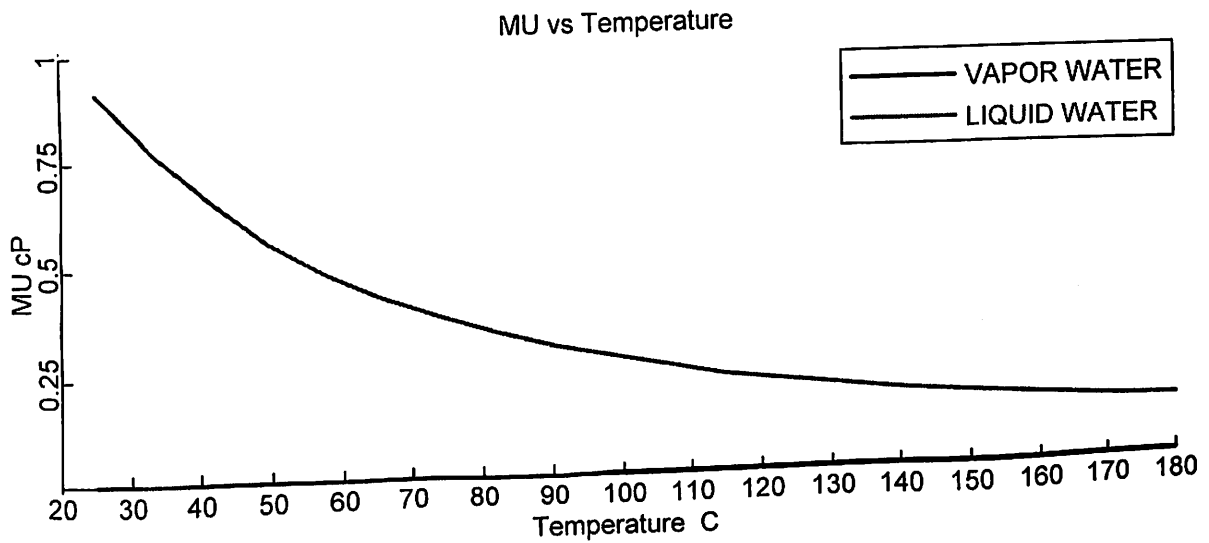


Fig.3.14 Viscosity of Water Vs Temperature

4.3 Specific heat:

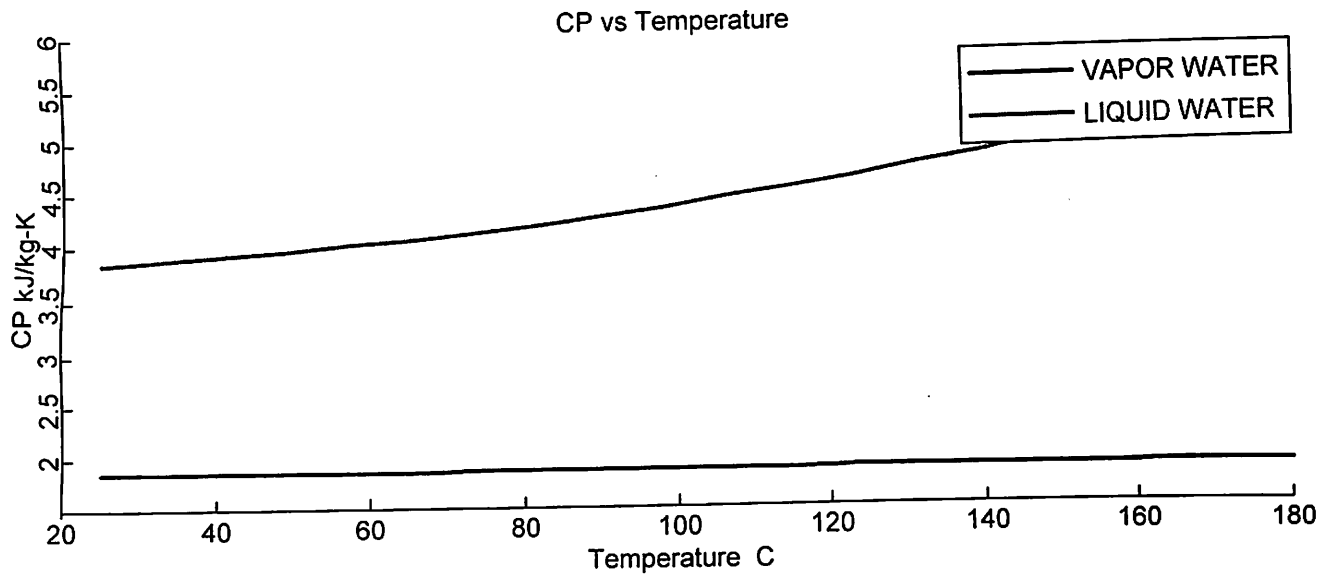


Fig.3.15 Specific Heat of Water Vs Temperature

4.4 Thermal Conductivity:

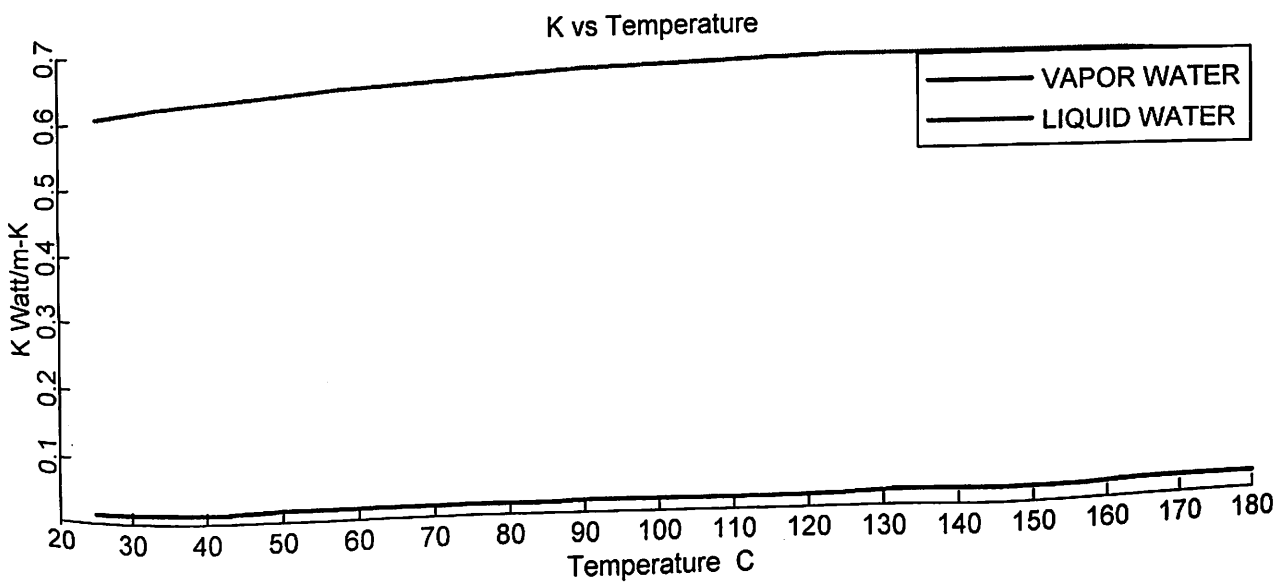


Fig.3.16 Thermal Conductivity of Water Vs Temperature

5. EDTA:

5.1 Density:

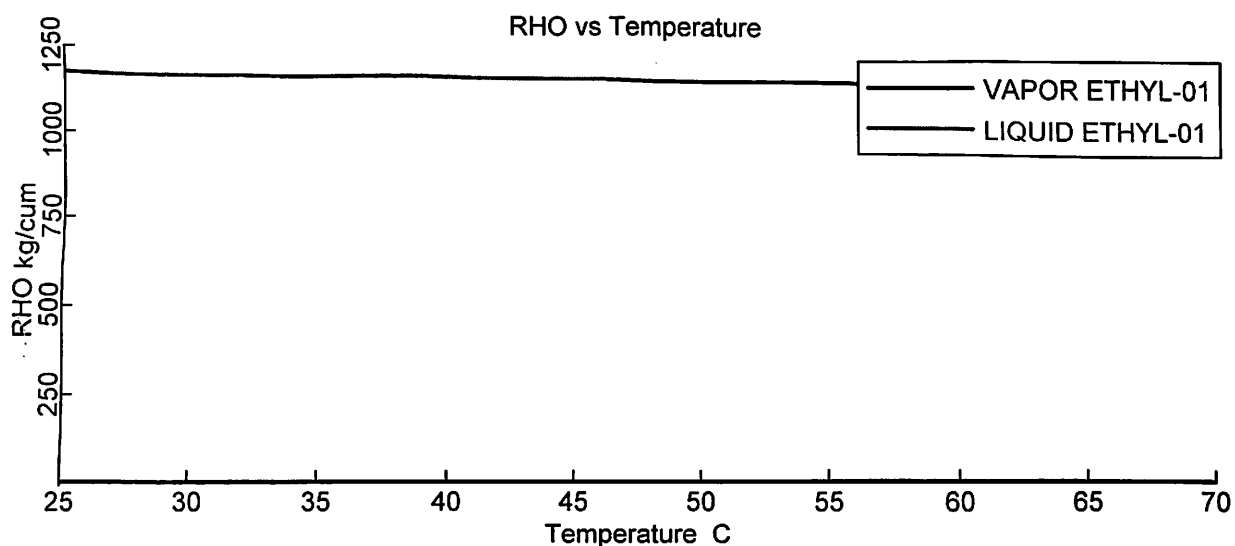


Fig.3.17 Density of EDTA Vs Temperature

5.2 Viscosity:

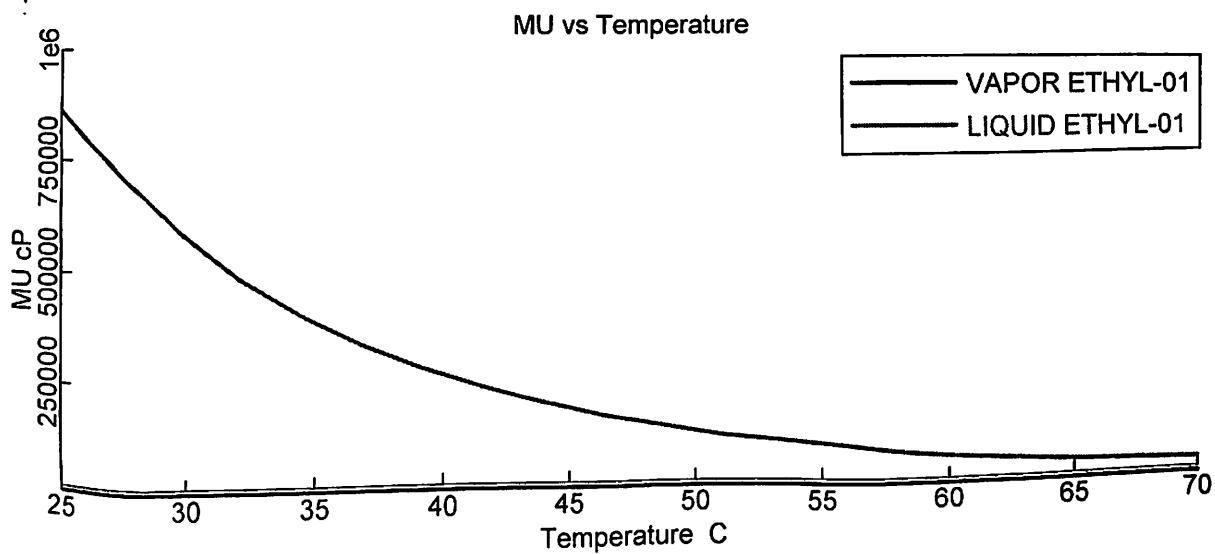


Fig.3.18 Viscosity of EDTA Vs Temperature

5.3 Specific Heat:

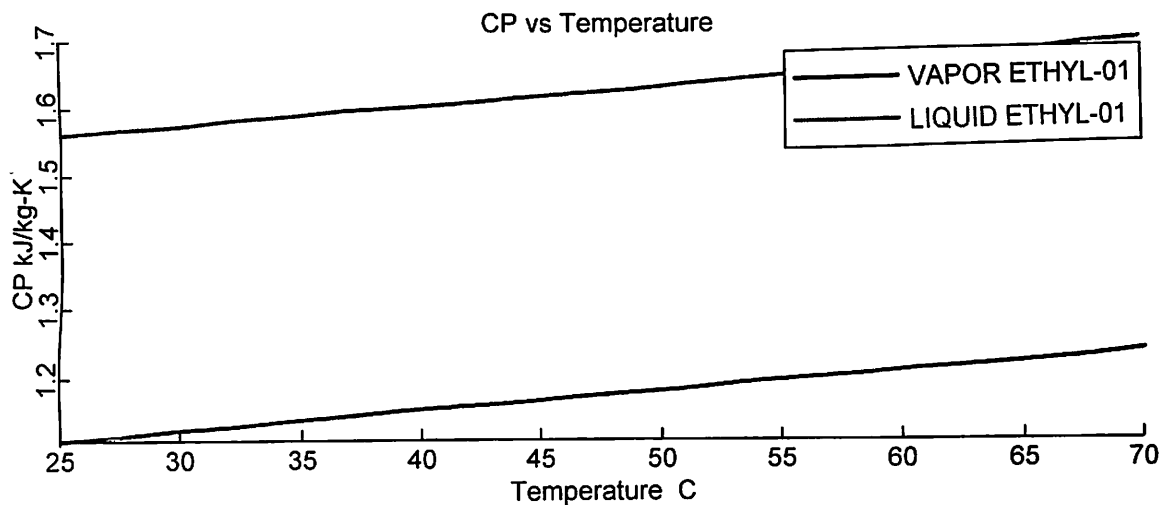


Fig.3.19 Specific Heat of EDTA Vs Temperature

5.4 Thermal Conductivity:

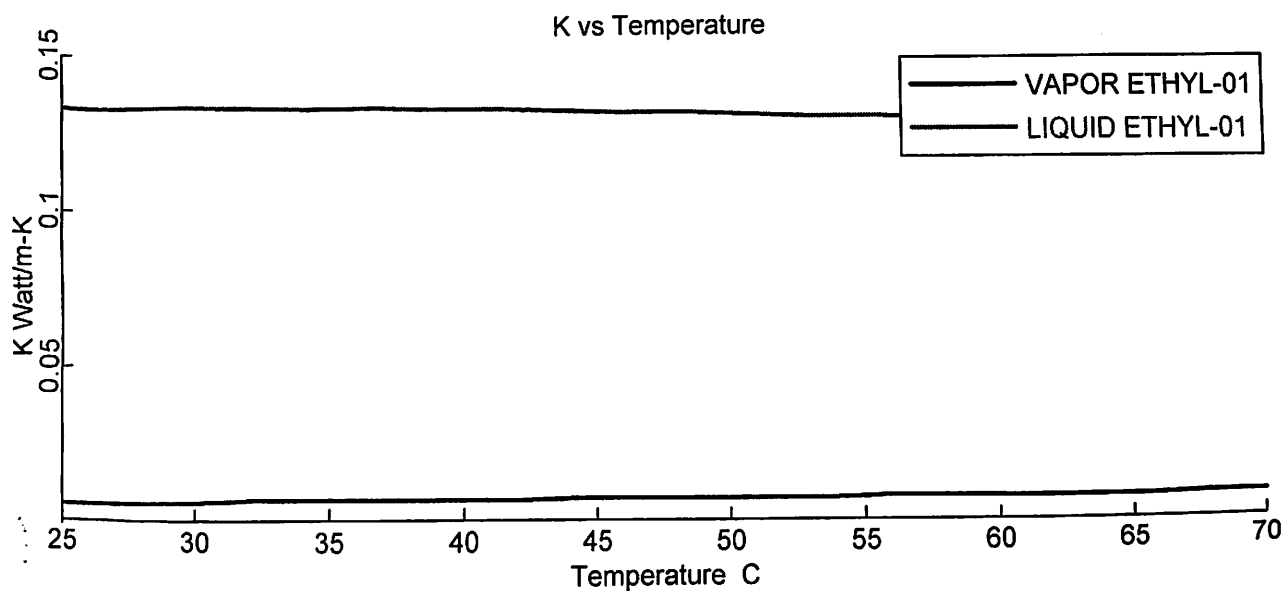


Fig.3.20 Thermal Conductivity of EDTA Vs Temperature

6. Methanol:

6.1 Density:

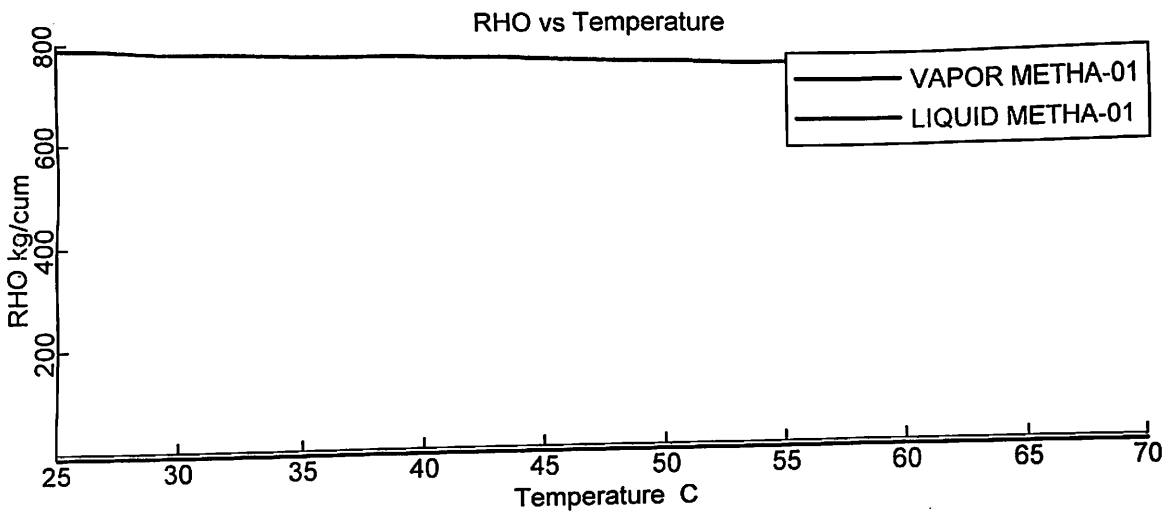


Fig.3.21 Density of Methanol Vs Temperature

6.2 Viscosity:

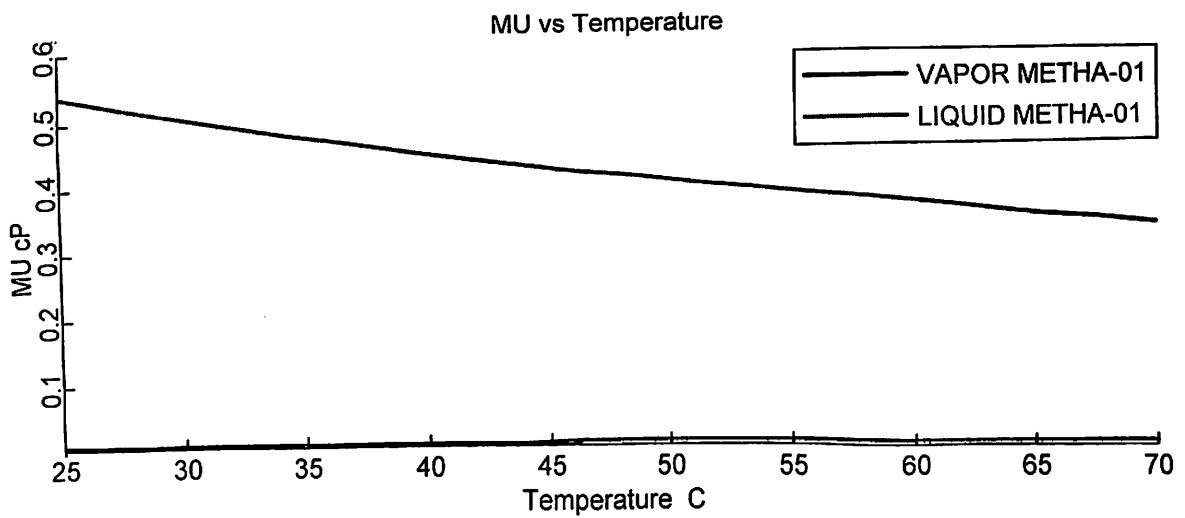


Fig.3.22 Viscosity of Methanol Vs Temperature

6.3 Specific Heat:

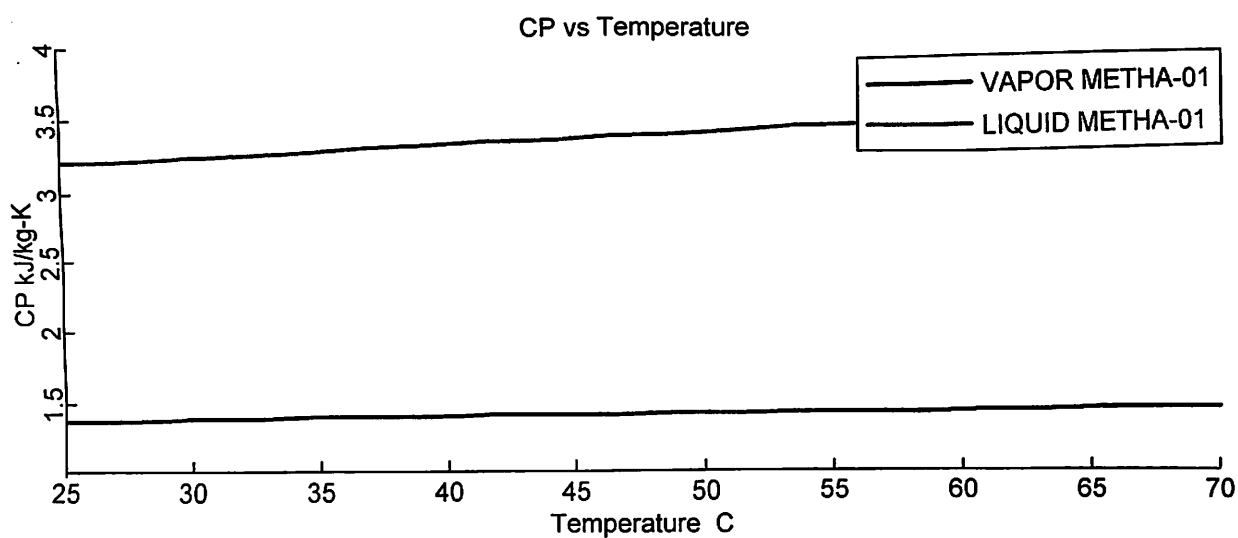


Fig.3.23 Specific Heat of Methanol Vs Temperature

6.4 Thermal Conductivity:

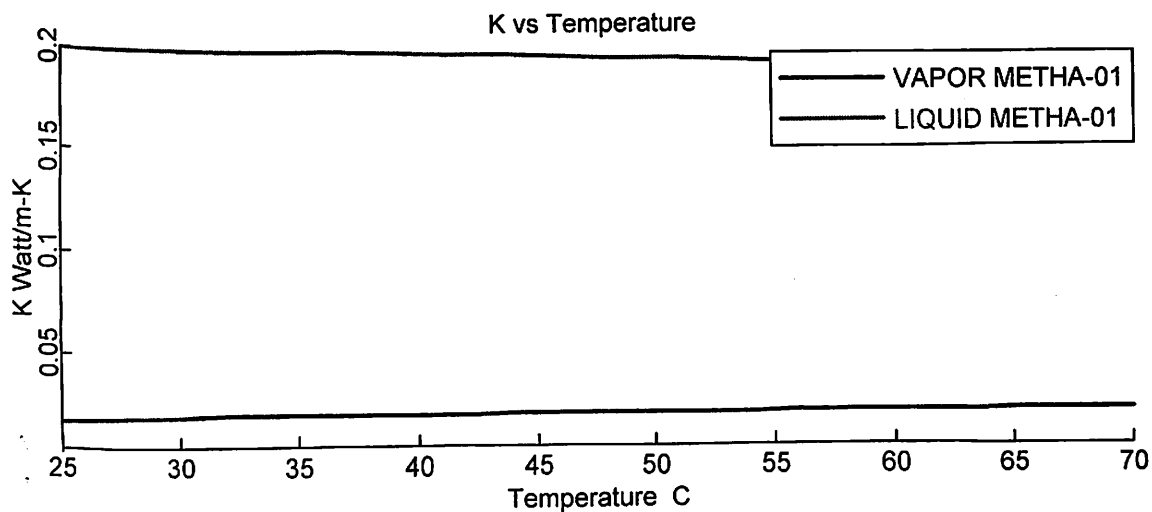


Fig.3.24 Thermal Conductivity of Methanol Vs Temperature

APPENDIX 4: Project Timeline

Month	Task Completed
March	Proposal and Acquisition
April	Approval from IICT
May	Training on ASPEN-Plus, MATLAB, etc.
June	-do-
July	-do-
August	Allotment of Project
September	Literature Review and Data Collection
October	Package selection and Base case
November	Simulation and Modelling
December	PFD, P&ID, ULD etc. of Pilot Plant
January	Joining IICT; Designing of allotted equipment
February	Design of Equipment
March	-do-
April	Design of Piping System, Submission of first draft, final report and Presentation of Seminar