# University of Strathclyde

## Department of Chemical and Process Engineering

# Development of an experimental arrangement for measurement of evaporation through measurement of changes in flowrate

By

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**A thesis presented in the fulfilment of the requirements for the degree of Master of Philosophy**

**2013**

# Declaration

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# Acknowledgements

I will like to express my thanks and gratitude to Dr. Mark Heslop for dedicating his time to the supervision of this thesis. I very much appreciate the informal discussions that we had throughout the length of project. Thank you for your patience and help and may Allah blessings be yours in abundance.

I also wish to express my deep appreciation to the teaching staff of ‘Department of Chemical and Process Engineering’ for their efforts in helping the students to achieve their goal.

It is the time to express my deepest gratitude towards my friends specially Abuzar, Aman Khan, Bilal Ahmad, Javed Khan, Sajid Ahmad, Hussain Khan and Rashid Ahmad. My stay in Glasgow would never have been so wonderful without their company.

A special word of thanks goes to the members of my family specially my father (Muhammad Ismail) who believed in my academic abilities at a very young age, thank you for your encouragements that kept me going.

Finally, thanks to the ‘Faculty of Engineering’ and ‘Department of Chemical and Process Engineering’, University of Strathclyde for the financial support which allowed me to complete my MPhil.

# Abstract

A Vaporiser is a device that delivers a required concentration of vapour into a gas stream. There are different types of vaporisers and also a range of methods for the measurement of evaporation rate. The existing measurement techniques include change in volume, mass and concentration.

This work is focused to measure the evaporation rate of vapours (ethanol) into a gas stream (nitrogen) through a new developed experimental method and also using the theoretical method.

In this work, a diffusion cell is developed at a small scale which would give a flow of ethanol vapours in a controlled manner. A microplant is also developed in a twin sided mode which consists of a differential pressure transducer (DPT), resistance tubing and empty tubing to measure extremely small changes in flow rate. The diffusion cell is then integrated with the microplant technology in which case the vapours (ethanol) are added from the diffusion cell into the main stream of gas (nitrogen). When vapours (ethanol) are added into main stream of nitrogen gas this resulted a step increase in the differential pressure transducer signal which is due to an increase in the resulting flowrate. The evaporation rate is then calculated from this change in flowrate. These results are then compared with the results achieved from theoretical method.

The results achieved from theoretical method indicated that the evaporation rate of ethanol is 0.052ml/min which is 3.2 times greater than the experimental diffusion rate (0.0163ml/min). This shows that diffusion rate in the experimental method is extremely low this is due to the reason that a conical flask and small diameter diffusion tube were used in the experimental setup which was not producing sufficient and continuous flow of vapours (ethanol).

The evaporation rate in the experimental method can be further increased by applying design changes to the current diffusion tube i.e. increasing its diameter and reducing the distance from the liquid surface and the main stream of nitrogen gas.

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# List of Symbols

| | |
|---|---|
| $A$ | Cross sectional area, (cm$^2$) |
| $B$ | Psychromatric constant |
| $C_A$ | Concentration of A, (moles of A per unit volume) |
| $C_P$ | Specific heat at constant pressure, (J/g.°C) |
| $D$ | Diffusion coefficient at pressure $p$, (cm$^2$/sec) |
| $D_{AB}$ | Diffusivity or diffusion coefficient for A in B |
| $D_{BA}$ | Diffusivity or diffusion coefficient for B in A |
| DPT | Differential Pressure Transducer |
| $h_{fg}$ | Latent heat of vapourisation, (J/kg) |
| $h$ | Enthalpy, (J/g) |
| $L$ | Length of capillary, (cm) |
| $M$ | Molecular mass, (g/mol) |
| MT | Microplant Technology |
| $n$ | Number of moles |
| $N_A$ | Molar flux of A, (moles per unit area per unit time) |
| $P$ | Atmospheric pressure, (Pa) |
| p | Partial pressure, (Pa) |
| $P$ | Total pressure, (Pa) |
| $P_L$ | Saturated vapour pressure of liquid, (Pa) |
| $r$ | Diffusion rate, (g/sec) |
| $R_a$ | Gas constant for air, (cm$^3$Pa/molK) |
| $R$ | Gas constant, (cm$^3$Pa/molK) |
| $T$ | Temperature, (°C) |
| $t$ | Time, (sec) |
| $T$ | Absolute temperature, (K) |
| $W$ | Humidity ratio |
| $y$ | Distance in the direction of transfer |
| $\Delta$ | Slope of saturation pressure line |
| $\gamma$ | Relative humidity, fraction |
| $\upsilon$ | Specific volume, (m$^3$/kg) |

| | |
|---|---|
| $\pi$ | Pressure term, $(P/(P-P_L))$ |

Subscripts

| | |
|---|---|
| a | Dry air |
| dp | Dew point |
| s | Condition at saturation |
| w | Condition for water vapour |
| wb | Wet-bulb |

# Chapter 1 Introduction

## 1.1 Theory of diffusion

Molecular diffusion can be defined as the transfer of molecules through a fluid by means of random motion of individual molecules. In general, this diffusion occurs in gas-gas, gas-liquid, liquid-liquid, gas-solid and liquid-solid and may also take place in solid-solid systems. In general, gases diffuse much faster than liquids and liquids much faster than solids. The rate at which diffusion occurs depends on a number of factors i.e. pressure, temperature and concentration gradients. For example, diffusion is more frequent in gases at higher temperature in which case the molecular velocities are greater and also at low pressure in which case the average distance between the molecules are greater and the number of collisions between the molecules are less frequent.

## 1.2 Methods for adding vapours into gas stream

The general methods for adding vapours into a gas stream are based on achieving either saturation conditions or a known rate of vapour diffusing into a diluent gas stream (Miguel and Natusch 1975). The following are two different methods through which vapours can be produced and added into a gas stream.

1. Anaesthetic vaporiser (saturated mixtures)
2. Evaporation of liquids in narrow tubes (dilute mixtures)

### 1.2.1 Anaesthetic vaporiser

Vaporiser is a device that delivers a required concentration of vapours which is usually an anaesthetic agent to a gas stream (air). The vapour in a vaporiser is made of molecules which have a high level of kinetic energy that has broken the bonds between their counterparts and escaped to the liquid surface. These molecules are in a state of random motion they leaves the liquid surface and also returns to it on a random basis and finally approaches towards an equilibrium at which point the vapour is fully saturated. These molecules exert pressure on the liquid surface and also on the sides of the container which at equilibrium is know as saturated vapour pressure. These molecules achieve high level of kinetic energy at higher temperature as a result of which more molecules leaves the liquid surface at which point the saturated vapour pressure increases. However, when the molecules having high level of energy escape the liquid surface it results in reducing the temperature of the liquid (Young et. al. 2010, Eales et. al. 2007, Gardner et. al. 1996).

In general, vaporisers are divided into two types, plenum and draw-over. There is a third type of vaporiser which is called a gas blender and applied specifically to deliver desflurane.

### 1.2.1.1 Plenum vaporiser

This type of vaporiser is driven by positive pressure. In this type of vaporiser the internal flow resistance is high therefore it required a pressurised source of gas to provide a constant flow of gas. Since the inlet pressure is constant plenum vaporiser deliver a constant concentration of anaesthetic agent over a wide range of gas flow. Figure 1.1 shows the working principle of plenum vaporiser.

The flow of gas is split in two streams, in this case a bypass stream (B) and the main stream which passes through the vaporising chamber (M) and saturates with vapours. The two flows are then mixed to get a final concentration of vapour in the gas stream is a function of the saturated vapour pressure and the ratio of M to B.

Figure 1.1 shows that if valve V1 is closed the gas will flow through a bypass stream in which case there will be zero concentration of vapours, conversely if valve V2 is closed and all the gas flow is channelled through the main stream then there will be maximum concentration of vapours. The vaporisation chamber wall is made of steel to provide and maintain a good thermal contact between the liquid and surroundings.

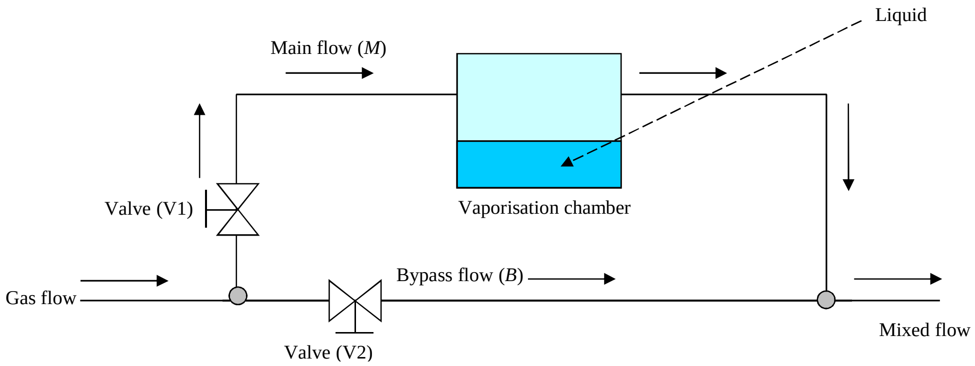

Figure 1.1 Working principle of plenum vaporiser

### 1.2.1.2 Draw-over vaporiser

This type of vaporiser is driven by negative pressure and has a low resistance to flow of gas. The working principle is shown in Figure 1.2 where the fresh gas is drawn by either a patient or a ventilator.

In this case the volume of air passing through the vaporising chamber is determined by the respiration rate of the patient. Figure 1.2 shows that air enters into the vaporising chamber because of the negative pressure created by the patient and then that air collects vapours from the vaporising chamber and finally delivers to the patient.

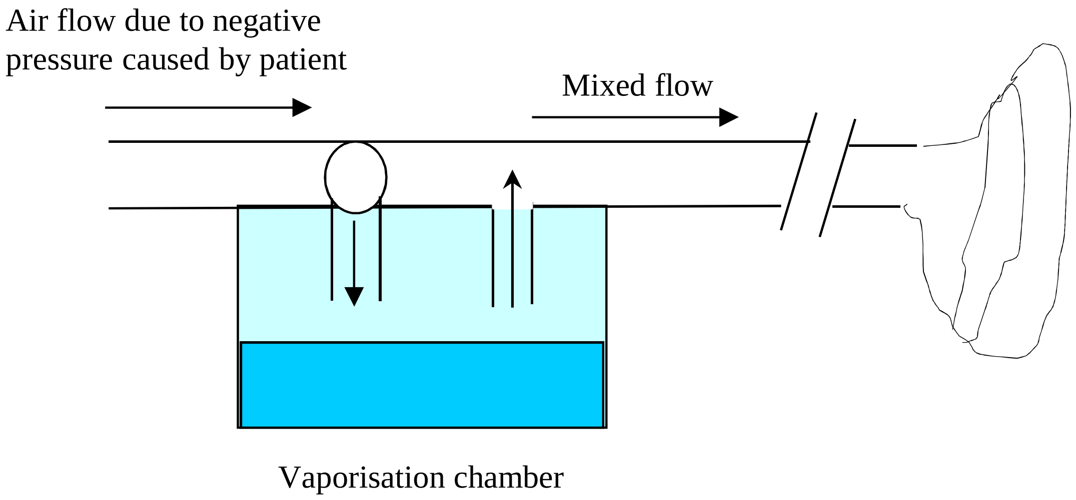

Figure 1.2 Working principle of draw-over vaporiser

#### 1.2.1.3 Problems with vaporizer design

The following are some of the problems which can be noticed in above vaporizers.

##### 1.2.1.3.1 Temperature control

The temperature of the liquid in the vaporization chamber may fall due to the escape of molecules having greater kinetic energy as a result of which the saturated vapour pressure may fall. It is therefore necessary to provide good thermal contact between the liquid and surroundings which can be achieved through the use of a metal container having thick walls to ensure good thermal contact between the liquid and surroundings. This would mean that heat is conducted from the surroundings through the metal wall into the liquid container, however in this scenario the surrounding temperature is likely to be a factor.

##### 1.2.1.3.2 Changes in flow

It is required that the concentration of vapour in the outlet stream is not affected by variation in gas flow rate. However, the vaporisation process is not likely to catch up at higher flowrate of incoming fresh gas at which point the amount of vaporised anaesthetic agent is inadequate.

The design of vaporiser has had been developing to achieve its ideal properties and the future vaporisers are likely to be more dependent on computer technology instead. The ideal properties of a vaporiser include low resistance to flow, lightweight, hard-wearing economic, minimal servicing, corrosion resistance and safe to use. In addition, the performance of a vaporiser should not be affected by the following parameters: fresh gas flow, volume of agent, temperature of both agent and ambient, both ambient and back pressure (Young et. al. 2010, Eales et. al. 2007).

### 1.2.2 Evaporation of liquids in narrow tubes

In this method the gas is passed over the top of the diffusion tube containing the volatile liquid. The temperature of tube and liquid is maintained constant and gas is passed over at the top of the tube at a rate to prevent turbulence (at the top of the tube). The following Figure 1.3 shows a very simplified diagram of such system where Liquid (A) is vaporising into a gas stream (B). This method can be used to produce extremely dilute gas mixtures e.g. 0.1 to 100ppm (Mckelvey and Hoelscher 1957).

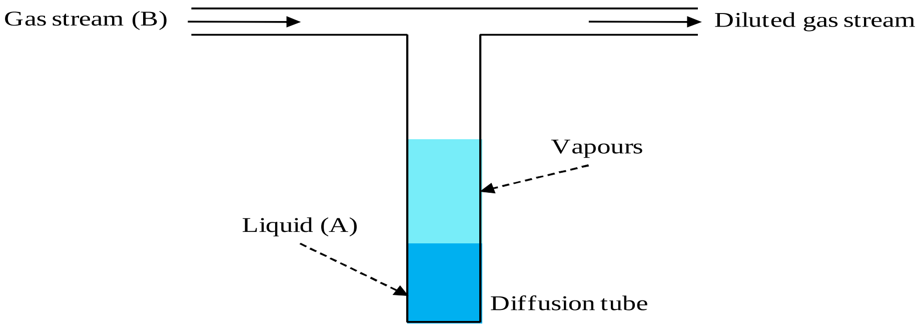

Figure 1.3 Evaporation rate through simplified diffusion tube

## 1.3 Measurement of evaporation rate

The evaporation rate can be measured by the following three different methods:

### 1.3.1 Change in volume

In this method, a known volume (initial) of liquid is taken and the gas is passed over the surface of the liquid. The liquid level falls due to evaporation of volatile components and doesn’t remain constant. The final volume of liquid is then measured and the difference between initial and final volume is divided by the total time of experiment which would then give the rate of evaporation in terms of volumetric flowrate.

### 1.3.2 Change in mass

The diffusion rate can also be determined by weighing the diffusion cell on an analytical balance before and after a run. This would then give the diffusion rate in terms of mass flowrate.

### 1.3.3 Change in concentration

The concentration of trace gases in the atmosphere vary from few ppt to several ppb. The technique used to measure the concentration of these traces gases (volatile organic compounds) in the atmosphere is gas chromatography combined with different detectors. These detectors produce an output signal which is a function of the concentration or mass of the sample being analysed. Gautrois and Koppmann (1999) developed a diffusion device which produces ambient air samples where the concentration of the trace component is between several ppt (v/v) and ppb (v/v). They analyzed the gas mixture samples with a gas chromatography combined with FID and ECD system in series which is used for the measurement of air samples.

## 1.4 Objectives of current research

The overall aim of current research is to design and develop a vaporiser that could give a constant and continuous flow of vapours into a gas stream at normal temperature and atmospheric pressure. This vaporiser or in other words diffusion cell would be then integrated with MT to measure the evaporation rate through change in flowrate.

The aim here is to measure the evaporation rate of vapours into a gas stream using the experimental method and also the theoretical method using the modelling equation for diffusion rate. The main motivation behind this research is that it provides an opportunity to extend our measurement techniques to vapour/liquid system. Secondly, it would enable us to achieve vapours at a very low concentration (at around 100ppm level) in a fresh gas stream at normal temperature and atmospheric pressure. In the current application, ethanol is selected (due to its high vapour pressure) as a volatile liquid which is diffusing into a nitrogen gas stream.

In future, this method could be further explored and refined and an alternative moisture measurement device could be developed. The gas carrying vapours stream could be passed through a solid desiccant which will absorb the vapours from the gas stream and as result would reduce the gas/vapours stream flowrate. The concentration of vapours in the gas stream is then calculated from this reduction in flowrate.

# Chapter 2 Literature Review

## 2.1 Introduction

In this chapter the background behind the diffusion/evaporation rate and its determination using the theoretical and experimental methods are discussed. The importance of moisture measurement and the various techniques used in the industry along with its limitations and operating ranges are also discussed. The important psychrometric properties and its relevant mathematical formulations are mentioned. Some of the important properties of ethanol which can be useful in the current work are also listed. To remove the moisture content in a gas stream the different types of solid desiccants and its selection criteria are also discussed. At the end of this chapter, the concept and application of MT is explored and its scope in the current research is also discussed.

## 2.2 Diffusion rate

This research is mainly focused on the gas-liquid systems where a volatile liquid is evaporating into a gas stream. The diffusion rate of a liquid vaporising into a gas stream can be determined either experimentally by keeping the diffusion tube/cell on a micro balance measuring the loss in weight after a period of time. The second method is theoretical which involves using the following modelling equation for diffusion rate for the steady state diffusion of one gas through a second stagnant gas (Sherwood et. al. 1952).

For example, if a diffusion tube is partially filled with volatile liquid A and which is surrounded by an insoluble gas B then liquid A vaporises and diffuses into gas B. This evaporation or diffusion rate is described by Fick's first law which represents the mass transfer rate as a linear function of the molar concentration gradient.

Assume that in the below Figure 2.1 ʎ is the mean distance a molecule travels in Z direction across plane PQ (Sherwood et. al. 1952).

The total moles of component A cross plane PQ from left to right is equal to $\frac{\lambda}{2}S\left(C-\frac{\lambda}{2}\frac{\partial c}{\partial Z}\right)$.

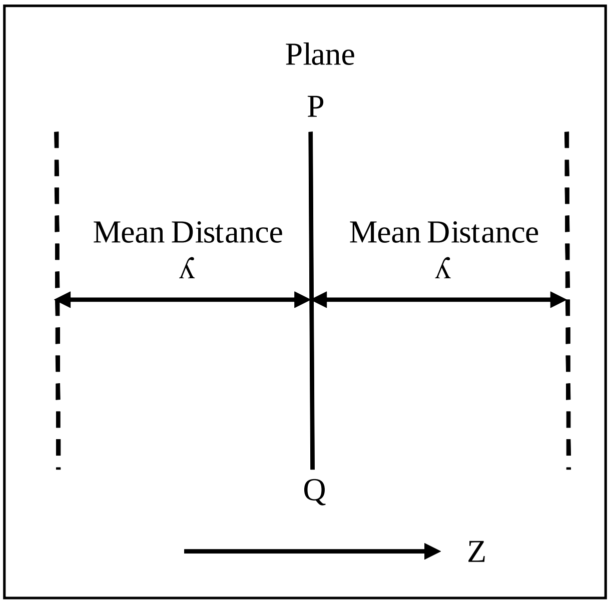

Figure 2.1 Explanation of Ficks Law

Here, S is the area of the plane PQ and C is the concentration of gas A at plane PQ. The mean distance travelled is ʎ, requiring a total time ʎ/u where u is the velocity.

The net rate of transfer of A is given by:

$$N_{AS} = -\frac{(\lambda\lambda S/2)(\frac{\partial C}{\partial Z})}{\lambda/u} = -\frac{\lambda u S}{2}\frac{\partial C}{\partial Z} \qquad 2.1$$

$N_A$ is the rate of diffusion of component A expressed as moles per unit time per unit area.

According to gas laws, the mole concentration is given by:

$$C = \frac{p_A}{RT} \qquad 2.2$$

Where $p_A$ is the partial pressure of gas A.

Equation 2.1 becomes

$$N_A = -\frac{\lambda u}{2RT}\frac{\partial p_A}{\partial Z} \qquad 2.3$$

Now handling only the case of equal molal diffusion of components A and B in opposite directions at constant $T$ and *ʎu* then $N_A = -N_B$ and $p_A + p_B = P$ where P is the total pressure.

For small element of volume of gas mixture, input must equal to output plus accumulation.

$$\frac{\partial C_A}{\partial \theta} + \frac{\partial N_A}{\partial Z} = 0 \qquad 2.4$$

Where $\theta$ is the time. Equations 2.3 and 2.4 can be combined and the simplified form is given below:

$$\frac{\partial p_A}{\partial \theta} = -\frac{\lambda u}{2}\frac{\partial^2 p_A}{\partial Z^2} \qquad 2.5$$

*ʎ* and $u$ are functions of the properties of the gas mixtures and the product *ʎu/2* may be replaced by $D_v$ which is termed as the diffusivity or diffusion coefficient for the pair of gases.

$$\frac{\partial p_A}{\partial \theta} = D_v\frac{\partial^2 p_A}{\partial Z^2} \qquad 2.6$$

This Equation is for equal molal diffusion of two gases in opposite directions and not for the diffusion of one gas through a second stagnant gas.

The steady state diffusion can be described as follows:

The total bulk flow of gas in the Z direction is $N_A + N_B$, the net transfer of component A is the rate of diffusion plus the transfer of A due to the bulk flow

$$N_A = -D_v\frac{dC_A}{dZ} + (N_A + N_B)\frac{p_A}{P} \qquad 2.7$$

(By putting $C = p/RT$ and then integrating the above equation)

$$\frac{D_v P}{RTZ} ln\left(\frac{1-\left(1-N_B/N_A\right)\frac{p_{A2}}{P}}{1-\left(1+N_B/N_A\right)\frac{p_{A2}}{P}}\right)=N_A+N_B \tag{2.8}$$

The steady state diffusion of one gas through a second stagnant gas,
Here, $N_B = 0$ the above equation can be simplified as follows:

$$N_A=-\frac{D_v P}{p_B}\frac{dC_A}{dZ}=-\frac{D_v P}{RTp_B}\frac{dp_A}{dZ}=\frac{D_v P}{RTp_B}\frac{dp_B}{dZ} \tag{2.9}$$

Integrating between the limits $Z_1$ and $Z_2$ in the direction of diffusion:

$$N_A=\frac{D_v P}{RT(Z_2-Z_1)} ln\frac{p_{B2}}{p_{B1}} \tag{2.10}$$

The logarithmic mean of the values $p_{B2}$ and $p_{B1}$ is given by:

$$p_{BM}=\frac{\left(p_{B2}-p_{B1}\right)}{ln\left(p_{B2}/p_{B1}\right)} \tag{2.11}$$

From Equation 2.10 and 2.11:

$$N_A=\frac{D_v P}{RTZ}\frac{\left(p_{B2}-p_{B1}\right)}{p_{BM}}=\frac{D_v P}{RTZ}\frac{p_{A1}-p_{A2}}{p_{BM}} \tag{2.12}$$

It is further simplified to give the below Equation:

$$N_A=\frac{D_v P}{RTZ} ln\pi \tag{2.13}$$

Here, $N_A = n/(A\times t) = m/(M\times t\times A)$

In Equation 2.13 the value of $N_A$ is putted and then simplified to give the below Equation:

$$\frac{m}{t} = \frac{D_v PMA}{RTZ} ln\pi \qquad 2.14$$

(Let $r = m/t$) then Equation 2.14 can be written as follows:

$$r = \frac{D_v PMA}{RTZ} ln\pi \qquad 2.15$$

Where $r$ is the diffusion rate (g/sec).

## 2.3 Diffusion tube/cell design

The idea of diffusion tube/cell has had been used in the past for a variety of applications. The literature study indicates that there is no work done on the diffusion cell/tube specifically for ethanol vaporising into nitrogen gas stream. However, the work done on other systems is discussed to get an idea of diffusion cell more specifically the dimensions of the diffusion tube. Therefore, in this section in most of the cases emphasis will be given on the design of the diffusion tube rather than the ultimate objective of the work done in the past.

Lee and Wilke (1954) developed an experimental arrangement to measure the vapour diffusion coefficient. Interestingly they also determined the diffusion coefficient for air-ethanol system which is 0.135cm$^2$/sec. Though the experimental arrangement is complex in nature and consists of many units, however the diffusion tube used in this setup has dimensions of 1.75cm diameter and 17cm length. In this experimental arrangement the diffusion tube dimensions are selected on a random basis, however it is useful to have reasonably large diameters to ensure the weighable evaporation loss in a rational time period without the extreme interruption by the gas flow inside the tube.

Mckelvey and Hoelscher (1957) designed the diffusion cell for preparation of dilute mixture of toluene in air. The design consists of using two 50ml round bottomed flasks connected by a straight glass diffusion tube which has diameter of 0.49cm and length of 10cm.

Altshuller and Cohen (1960) applied the concept of diffusion tube/cell for the production of known concentrations of gaseous hydrocarbons. They developed two cells where the diffusion tube diameters are in the range of 11.07 to 11.08mm and lengths of the tube are between 12 to 14cm. They also recommended that the cross sectional area of the diffusion tube should be uniform.

Miguel and Natusch (1975) developed a diffusion cell for the preparation of dilute vapour concentration where the vapour concentration ranging from few ppm to several thousand ppm can be obtained. In this application the diffusion tube having diameter of 1.1cm and length 8cm is used.

Gautrois and Koppmann (1999) used the diffusion method for the production of gas standards for the calibration of gas chromatographic measurements ambient air samples. The experimental setup consists of a diffusion vial (4ml) and a stainless steel diffusion tube of 1mm diameter and lengths between 10-100mm which depends on the diffusion coefficient and vapour pressure of the compound.

The above study doesn’t provide any information for ethanol vaporising into nitrogen gas stream, however it provides confidence in using the diffusion cell for such systems. In addition, it also gives an idea in terms of the diffusion tube dimensions for the current application.

## 2.4 Ethanol properties

Ethanol also called ethyl alcohol is a volatile, flammable and colourless liquid. The following are the main properties of interest in current work. Some of the important properties considered during this work are listed in Table 2.1 (Wikipedia, Lee and Wilke, 1954).

Table 2.1 Properties of ethanol

| | |
|---|---|
| Molecular formula | $C_2H_6O$ |
| Molar mass | 46.07g/mol |
| Density (liquid) | 0.789g/cm$^3$ |
| Boiling point | 78°C |
| Melting point | -114°C |
| Saturated Vapour pressure (@20°C) | 43.5mmHg |
| Viscosity | |
| (@25°C) | 1.074cp |
| (@20°C) | 1.773cp |
| Vapour Diffusivity (Ethanol-Air system) | 0.135cm$^2$/sec |

## 2.5 Humidity sensors

Humidity is very familiar term in the literature and can be defined and recognised as the presence of water vapour in air or any other gas. Its detection and control is becoming more and more important not only for industrial processes but also for human comfort.

This concept of humidity and its measurement techniques are reviewed here because the ultimate objective of this research would be to find the possibility of an alternative method for measuring moisture content in a gas stream by measuring the change in flowrate using the chemical engineering concept of flowrate measurement and material balances.

In 1986 Yamazoe and Shimizu classified the humidity sensitive materials into the following three main groups**:**

1. Electrolytes
2. Organic Polymers
3. Porous Ceramics

The humidity sensor that can be used for a range of applications should keep combination of the following properties: good sensitivity in wide range of humidity, quick response, good reproducibility, no hysteresis, fitness to circuitry, tough durability and reliability, resistance to contaminants, insignificant dependence on temperature, simple structure and Low cost.

### 2.5.1 Techniques for humidity detection

There are several methods to measure humidity in a gas stream. Some of the important detection techniques, their applications, limitations and dew point ranges are discussed here.

#### 2.5.1.1 Mechanical hygrometer

This is one of the oldest techniques for humidity detection which is based on the use of a material which expands and contracts with change in humidity. The most common materials used in this technique are human hair and synthetic fibres. This method is very simple and also inexpensive, however it is very slow and gives non-linearity and hysteresis issues which makes it unsuitable for applications where environmental conditions change rapidly (Visscher 2000, Car-Brion 1986).

#### 2.5.1.2 Chilled mirror hygrometer

This device is also known as condensation dew point hygrometer which is based on the optical technique to determine the dew point temperature. This type of hygrometer contains a temperature controlled reflective condensation mirror and an optoelectronic module. In operation, a sample of gas is usually passed through mirror, the mirror is cooled in a controlled manner until dew or frost is detected at which point the optical signal is reflected which is monitored by the optoelectronic module. The overall range of this type of hygrometer is -80°C to +100°C dew point. The highest accuracy is expected to be 0.03°C and 0.05°C in the range of -20°C to +40°C (Visscher, 2000). The accuracy of chilled mirror hygrometer is quoted to be as high as ±0.1°C (Wiederhold, 1997). The advantages of this method are wide range of dew points and also provide accurate and reliable measurements. The disadvantage of this method is that it requires regular maintenance due to the susceptibility of the mirror to contaminants.

#### 2.5.1.3 Wet and dry bulb psychrometer

This method consist of two thermometers, one of which is covered with a damp wick to determine the wet bulb temperature and the other measures the dry bulb temperature of the sampled gas. Once the wet and dry bulb temperatures are known then the humidity can be found through psychrometric charts and equations. This type of technique using thermometers with a temperature accuracy of ±0.2°C offers humidity accuracy of ±3%RH when operating over a temperature range of 5°C to 80°C (Wiederhold, 1997).

This is a simple and low cost method, however there is a chance of error in measurement because of the large number of variables that can affect the results and should be controlled for accurate measurements.

#### 2.5.1.4 Electrolytic hygrometer

The electrolytic sensors works on the principles of Faraday's laws of electrolysis to determine the moisture content in the gas stream at very low PPM range. The cell of electrolytic sensor is coated with a thin film of phosphorous pentaoxide ($P_2O_5$) which is an extremely hygroscopic material that absorbs water from the sampled gas at which point the hydrated pentaoxide becomes conducting and electrolyzes the water molecule into $H_2$ and $O_2$ due to the voltage difference between electrodes. The amount of current consumed in this process to dissociate the water molecule is proportional to the amount of water content in the sampled gas stream.

The range of this type of sensor is 1 to 2000 ppmv with ±5% measurement accuracy (Wiederhold, 1997). The range and accuracy is quoted to be as high as 0-3000 ppmv and ±2% respectively (Carr-Brion, 1986). These types of sensors have low limits of detection and also don't require any calibration. However, some of the disadvantages includes constant gas flow rate, high reactivity of pentaoxide which limits the range of gases, cell is destroyed by accidental water immersion and also regular regeneration of cell (Carr-Brion, 1986).

#### 2.5.1.5 Lithium Chloride (LiCl) hygrometers

This type of hygrometer consists of two metal electrodes coated and separated by LiCl and the leads of the electrodes are connected to an alternating voltage source. The moisture in the gas stream is absorbed by LiCl which makes it a conductor that allows the flow of current through it. The flow of current heats up the sensing element at which point water evaporates from the salt and its conductivity drops. This process continues to point where equilibrium is achieved and it neither takes nor gives off water. This equilibrium temperature is measured using platinum resistance thermometer and which is proportional to the water vapour pressure or dew point. The humidity range is between 10 and 100% at 20°C, with a dew point range of -40°C to 90°C and the accuracy is ±0.5°C (Carr-Brion, 1986).

These sensors are simple, low cost, durable and also provides measurements of both dew point and relative humidity. However, its response is very slow and can't measure very low moisture concentrations which limits it use for variety of applications more specifically for applications where very low moisture measurements is required.

#### 2.5.1.6 Aluminium oxide hygrometers

This is a capacitance type humidity sensor which is formed by depositing a layer of porous aluminium oxide on a conductive substrate and then coating the oxide layer with a water permeable but conductive thin gold layer. The substrate and thin gold layer are the first and second electrodes of the capacitor with the aluminium oxide as a dielectric. The water penetrates through the thin film of gold layer and is absorbed by the porous aluminium oxide layer. The amount of water absorbed is sensed electrically by measuring the change in capacitance which is then proportional to the water vapour pressure.

The standard sensors cover dew point between -110°C and +20°C which means a range of around 0.001ppm to 0.2% by volume. The accuracies range from ±1°C to ±2°C at higher dew points and ±2°C to ±3°C at -100°C (Carr-Brion, 1986).

These sensors operate over a wide range of temperature and pressure and have high selectivity for moisture, however the main disadvantage involved is that these sensors required frequent recalibration to accommodate ageing effect, contamination and hysteresis.

#### 2.5.1.7 Silicon hygrometers

This is also a capacitance type humidity sensor and somewhat similar to aluminium oxide sensors but using silicon as a sensor material instead. Its application range is broader than that of aluminium oxide sensors and would be preferred for most applications due to its enhanced characterisations.

The dew point range is between -80°C to above +80°C. These sensors also has a wide range from below 1.0 ppm to saturation, however they are not absolute sensors and required recalibration (Carr-Brion, 1986).

#### 2.5.1.8 Polymer humidity sensors

Polymer based humidity sensors are classified into two main categories i.e. resistive type and capacitive type (Sakai et. al., 1996). The resistive type respond to moisture variation by changing its conductivity and the capacitive type respond to water vapour by changing its dielectric constant. The operating temperatures range is between -50°C and 125°C and relative humidity ranges between 0.5% to 100%, accuracy depends on the humidity excursions e.g. an accuracy of around ±1% can be achieved with maximum excursions of 10-20% (Carr-Brion, 1986).

These are simple, low cost and give a rapid response to measure relative humidity, however they measure a limited range of moisture content and gives hysteresis and drift issues at high humidities.

### 2.5.1.9 Ceramic humidity sensors

These are similar to polymer type sensors in application and performance, however it shows enhanced performance at high temperature and also offers good resistance to some inorganic vapours. A porous ceramic sensor has been developed to measure the concentration of moisture in gases in the range of 50-100 ppmv (Basu et. al., 2001).

However, these type of sensors show very limited response at very low moisture concentrations and also sensitive to surface deposition.

### 2.5.1.10 Infra-Red (IR) moisture sensor

It works on the principle of dual wavelength absorption technique where one measures the amount of IR radiation at primary wave length at which point strong optical radiation absorption is observed and the other at reference wave length where no or very little absorption takes place. Humidity is then measured in the form of transmission ratios at primary and reference wavelengths. The range of concentration that can be measured is between few ppm up to 100% with overall accuracy of ±1%, however this is based on the gases used in operation and also chosen wavelengths (Carr-Brion, 1986).

This method provides direct measurements of water absorption and also covers a wide range of concentrations; however it is very expensive method and also requires adequate calibration to get accuracy of ±1% (Carr-Brion, 1986).

### 2.5.1.11 Crystal oscillator sensor

In this method the relative measurement of moisture content is done directly by measuring the changes in resonance frequency of hygroscopically coated quartz crystals. The change in humidity of the gas results a change in the resonance frequency of the quartz crystals which absorbs or desorbs water vapour because of the hygroscopic coating. The range of this measurement device is from below 0.02 ppmv up to 100,000 ppmv with accuracy of 1ppm or 5% (Carr-Brion, 1986). This method covers a wide working range, however it very expensive and bulky and can’t be used in-stream.

#### 2.5.1.12 Fiber-optic techniques for humidity detection

The introduction of optical fibre technology has compelled the researchers to focus on fibre optic based techniques for humidity detection. These sensors show additional features like small size, immunity to electromagnetic interference, multiplexing and remote sensing capabilities as compared to electronic or mechanical hygrometers. The techniques include direct spectroscopic, evanescent wave, in-fibre grating and interferometric methods. An extensive review on these methods and their RH ranges has been conducted recently by Yeo et al. 2008.

The fibre-optic sensing technology provides another approach to measure the moisture content, however the operating ranges and accuracy are some of the factors that limit its use in a range of applications (Yeo et. al. 2008).

This shows that there is variety of techniques to measure the humidity or moisture content in a gas stream but none of these can offer a perfect solution to meet with the ever changing demand in the industry. The literature study indicates that capacitive and resistive based sensors are commonly used in different applications, however around 75% of the humidity sensors in the current market are based on capacitive techniques (Yeo et. al., 2008, Rittersma, 2002, Scholz, 1992).

## 2.6 Psychrometric properties

Psychrometry deals with the determination of the properties of gas vapour mixtures. In this section, the general definitions and equations used to find the psychrometric properties from any two independent psychrometric properties of an air-water vapour mixture along with the atmospheric pressure are presented (Singh et. al. 2002).

Please refer to Figure 7.1 in the Appendix for the psychrometric chart of air-water vapour system (Perry’s Chemical Engineering).

### 2.6.1 Ideal gas law

It represents the equation of state of a hypothetical ideal gas and which provides a good approximation to the behaviour of many gases under many conditions.

$$PV = nRT \tag{2.16}$$

### 2.6.2 Saturation vapour pressure

The saturation vapour pressure is an important parameter in calculating other psychrometric properties.

The following equations can be used for calculating the saturation vapour pressure at the mentioned specific temperature range:

$$P_{ws,T} = 610.78 exp\left[\frac{17.269T}{237.3+T}\right] \tag{2.17}$$

$$(0^{\circ}C < T < 63.0^{\circ}\mathrm{C})\ \text{and}\ (610.78\mathrm{Pa} < P_{ws,T}\ \text{or}\ P_w < 22870.52\mathrm{Pa})$$

The above equation can be used to calculate saturation vapour pressure as a function of temperature and also it can be used the other way round for calculating temperature as a function of saturation vapour pressure within the mentioned limits for temperature and pressure.

### 2.6.3 Humidity ratio

It is the weight of the water vapour in kg/kg of dry air and can be determined by the following equations.

$$W = \frac{Mass\ of\ water\ vapour\ (m_w)}{Mass\ of\ dry\ air\ (m_a)} = \frac{\left(PV/RT\right)_w}{\left(PV/RT\right)_a} = \frac{P_w R_a}{P_a R_w} = \frac{\left(R_a/R_w\right)P_w}{\left(P-P_w\right)} \tag{2.18}$$

(The values of V and T are same for water vapour and dry air)

$$W = \frac{0.62198P_w}{P-P_w} \tag{2.19}$$

Where, $\left( {R_a}/{R_w} \right)$ is the ratio of mole masses of air and water (0.62198).

Equation 2.19 can be rewritten for actual vapour pressure:

$$P_w = \frac{PW}{0.62198 + W} \tag{2.20}$$

### 2.6.5 Relative humidity

It is the ratio of actual vapour pressure to saturated vapour pressure at the same temperature and mathematically can be represented as follows:

$$\gamma = \frac{P_w}{P_{ws} T_a} \tag{2.21}$$

### 2.6.6 Dew point temperature

The dew point can be defined as the temperature where the water vapour in a volume of humid air at constant barometric pressure will condense into liquid water. The dew point temperature can be calculated by using saturation vapour pressure Equation (2.17) and putting the value of actual vapour pressure ($P_w$) or saturation vapour pressure at dew point temperature.

## 2.7 Solid desiccant dehydrator

Solid desiccant is an adsorption process in which case the water molecules are taken out from the gas stream and held on the solid surface. This is a physical phenomenon and doesn’t involve any chemical reaction unlike absorption process. The adsorption process depends on the operating temperature and pressure of the system, for example increasing the system pressure and decreasing the temperature the adsorption is likely to increase and vice versa (Petroleum Extension Service,1972). One of the main advantages of using solid desiccant is that lower dew points and be achieved over a wide range of operating conditions. Besides, there is no risk of corrosion or foaming in the system.

### 2.7.1 Properties of solid desiccants

There are some of the important properties that a solid desiccant should posses to achieve its desired target in the economical way (Kohl and Nielsen 1997).

- Large surface area for high capacity and high mass transfer rate
- High bulk density and activity for the components to be removed
- Must be easily and economically regenerated
- There should be little resistance to gas flow to lower the pressure drop
- Must have high mechanical strength to resist crushing and dust formation
- Must be economical, non-corrosive, non-toxic and chemically inert
- Must retain strength when get wet

### 2.7.2 Types of solid desiccants

The most commonly used desiccants for gas dehydration as follows (Campbell, 1984., Aitani, 1993):

#### 2.7.2.1 Alumina-based adsorbents

These type of adsorbents include impure naturally occurring materials such as bauxite and purity activated aluminas derived from gels/crystalline materials.

#### 2.7.2.2 Molecular sieves

This type includes synthetic zeolites with extremely uniform pore dimensions.

#### 2.7.2.3 Silica-based adsorbents

This includes activated silica gel and special formulations which contains a small fraction of other components.

### 2.7.3 Selection criteria

In general the selection of adsorbent for a particular operation is an economic exercise (Gandhidasan et. al. 2001). However, the current work looks at an adsorbent which can remove moisture content at a very low concentration in a gas stream. Therefore, based on the operating conditions, availability and requirement of the current work silica gel and molecular sieves are likely to be the competent adsorbents which can remove the moisture content at a very low level.

The effectiveness of adsorbent is described here just to show its suitability for the removal of water vapours from humid air. The carbon molecular sieves can adsorb substantial amount of water from the air stream i.e. in the range of 400-450mg per gram of adsorbent (F.Patrycja et. al 2005).

## 2.8 Application of microplant technology (MT)

Microplant technology (MT) is defined as the application of a DPT, resistance tubing and empty tubing to measure extremely small changes in flow rate. This technology has already been applied to a number of applications: measurement of the adsorption of binary gas mixtures on molecular sieves, measurement of reaction rates and conversions, measurement of viscosity of gas mixtures and measurement of volume of mixing (Heslop et. al. 2008, Heslop et. al. 2008, Heslop et. al. 1997, Mason et. al. 1998).

In this section, the concept of MT is introduced by defining the work of Mason, Buffham, Heslop and Zhang. They developed a Microplant to measure the changes in viscosity and flowrate. The following Figure 2.2 shows a very simplified diagram of their experimental setup. It consists of five components the main gas flow enters the upstream capillary choke A at constant pressure this is the point where most of the pressure drop occurs. The gas then passes through valve B and enters the delay line C where very little pressure drop occurs. After passing through the delay line the gas then passes through the downstream capillary choke E at which point only 1% pressure drop occurs and after which the gas then escapes to the atmosphere. The pressure transducer D measures pressure just upstream of the downstream choke E. The operating philosophy is such that initially at time zero gas is passed through the system and valve B is closed. When the pressure p (0) at D stabilizes as shown in Figure 2.3 then valve B is switched to add small flow of perturbation gas (around 1% of the main flowrate) into the main stream at which point the pressure sensed at D increases which is represented by p (1) in the

Figure 2.3 which in other words is the increase in flow. This new composition of gas when reaches the downstream choke E the pressure at D changes again at which point it represents the increase/decrease in viscosity as shown in
Figure 2.3 (Mason et. al. 1998).

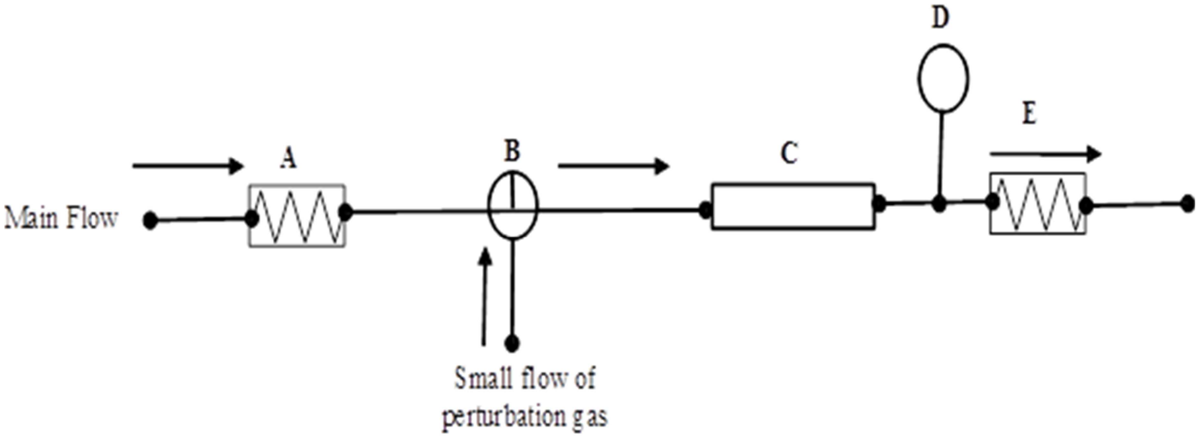

Figure 2.2 Simplified Microplant for flowrate and viscosity measurements

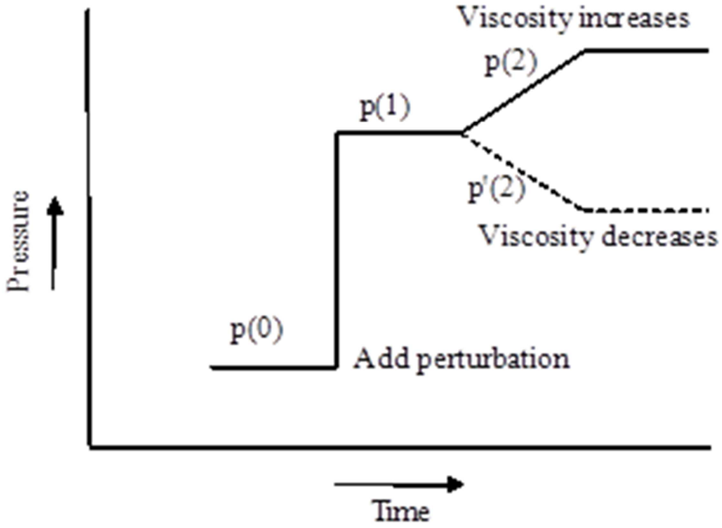

Figure 2.3 Response of pressure transducer with variation in flowrate and viscosity

### 2.8.1 Analysis

In Figure 2.2 initially when the capillary choke acts as a flowmeter then $p_1$-$p_0$ is proportional to the increase in the flowrate and later on when it behaves as a viscometer then $p_2$-$p_1$ is proportional to the change in viscosity.

The mathematical representation of the above is as follows:

$$\frac{dp_z}{dz} = -K_c \mu Q \qquad 2.22$$

Where, $Q$ is the volumetric flowrate, $\mu$ is the viscosity and $Kc$ is a constant depending on the characteristics of the tube.

For Ideal gas:

$$Q = \frac{MRT}{p_z} \qquad 2.23$$

Where, $M$ is the molar flowrate of gas, $p_z$ is the pressure at disctance $z$, $T$ absolute temperature and $R$ is the gas constant.

Combining the above Equations, the longitudinal pressure can be expressed as follows:

$$p_z \frac{dp_z}{dz} = -K_c \mu MRT \qquad 2.24$$

Integrating Equation 2.24 gives:

$$p^2 - P_{BPR}^2 = 2K\mu MRT \qquad 2.25$$

Where, $K$=$K_cL$, here $L$ is the length of the tube and $p$ is the pressure at the inlet of the downstream capillary choke. $P_{BPR}$ is the outlet pressure of the downstream of capillary choke.

The inlet pressure changes due change in flowrate and viscosity and this can be represented as follows:

If the inlet pressure is raised by $\delta p$ due to change in flowrate $\delta M$ and viscosity $\delta\mu$ then Equation 2.25 can be represented as follows:

$$(p+\delta p)^{2}-P^{2}{}_{BPR}=2K(\mu+\delta\mu)(M+\delta M)RT \qquad 2.26$$

Subtracting Equation 2.25 from Equation 2.26:

$$2p\delta p+(\delta\delta p^{2}=2K(\mu K(\quad +M\delta\delta+\delta M\delta\mu)RT \qquad 2.27$$

This is a second order Equation where the second order terms are small for small changes, therefore it is simplified further:

$$\frac{2p\delta p}{(p^{2}-P_{BPR}^{2})}=\frac{\delta M}{M}+\frac{\delta\mu}{\mu} \qquad 2.28$$

Using Equation 2.28 for first step change in Figure 2.3: $(\delta\mu=0)$

$$\frac{2p_0(p_1-p_0)}{(p_0^{2}-P_{BPR}^{2})}=\frac{\delta M}{M} \qquad 2.29$$

Using Equation 2.28 for second step change in Figure 2.3: $(\delta M=0)$

$$\frac{2p_1(p_2-p_1)}{(p_1^{2}-P_{BPR}^{2})}=\frac{\delta\mu}{\mu} \qquad 2.30$$

The ratio of Equation 2.29 and 2.30 results into the following Equation:

$$\frac{p_1(p_2-p_1)}{p_0(p_1-p_0)}\frac{(p_0^{2}-P_{BPR}^{2})}{(p_1^{2}-P_{BPR}^{2})}=\frac{M}{\mu}\frac{\delta\mu}{\delta M} \qquad 2.31$$

If the capillary and pressure ($p_0$, $p_1$, $p_2$) step sizes are selected such that $p_0 \approx p_1 \approx p_2$ and $p_0^{2}-P_{BPR}^{2} \approx p_1^{2}-P_{BPR}^{2}$.

$$\frac{(p_2 - p_1)}{(p_1 - p_0)} = \frac{M}{\mu}\frac{\delta\mu}{\delta M} \qquad 2.32$$

Please refer to Reference Mason.G et. al. (1998) for the very detailed description.

### 2.8.2 Scope of MT in current research

In this project, an effort is made to investigate whether the same Microplant could be modified and then integrated with a diffusion cell to measure the evaporation rate of ethanol into nitrogen gas stream. This research could be then further extended to develop an alternative detector for moisture content in a gas stream which could be achieved by developing an arrangement in which moisture is added to a dry gas stream, then passed through a suitable solid desiccant, and measure the reduction in flow rate - this reduction should then give the moisture content.

# Chapter 3 Methodology

## 3.1 Introduction

In this chapter the methodology behind developing the Microplant for steady baseline and then modifying it and adding a stream of a small flow of perturbation gas and also integrating it with the diffusion cell are discussed. The process flow diagrams and operating philosophy of experimental setup for baseline line, adding a small flow of perturbation gas (Nitrogen) and diffusion cell are also discussed.

## 3.2 Experimental arrangement for baseline

The following Figure 3.1 shows the experimental arrangement for the baseline. It consists of nitrogen cylinder which provides the main flow of nitrogen gas, Porter Mass Flow Controller (MFC, Type VCD1000), Porter Pressure Regulator (PR, range 0-60psi), Porter Back Pressure Regulator (BPR, range 0-30psi), High Resistance Block, Furness Control DPT (types: ±1000 mm $H_2O$), Low Resistance Block and a second Porter Back Pressure Regulator (BPR, range 0-30psi).

The system is arranged in a twin sided mode as shown in Figure 3.1, the main reason for which is to maintain a steady baseline from which to measure the changes in flow. The experimental setup consist of allowing the nitrogen gas to flow through at pressure regulator at which point the pressure is set to 40psi and then allowed to pass through a mass flow controller which controls the mass flow of nitrogen and can deliver a range of flow rates. This first downstream back pressure regulator maintains a constant upstream pressure and the excess from the mass flow controller is directed to vent through a rotameter. The flow is then equally divided and flows through brass block containing large flow resistances to set the flows and the purpose of block is to act as a thermal mass. The DPT measures the pressure difference between the main and reference side. Further downstream is the brass block small a flow resistance to measure the flows and the purpose of the block is to act as a thermal mass. The back pressure regulator at the end maintains a constant inlet pressure.

The connecting tubing is made of nylon (1/8inch) and the whole experimental setup is enclosed in a metal box to ensure that the system is thermally insulated.

The experimental data is measured using DPT ( type FC0332) supplied by Furness Controls with a range of ±1000mm$H_2O$. The experimental data is recorded using a Pico ADC-24 (24bit) data acquisition system.

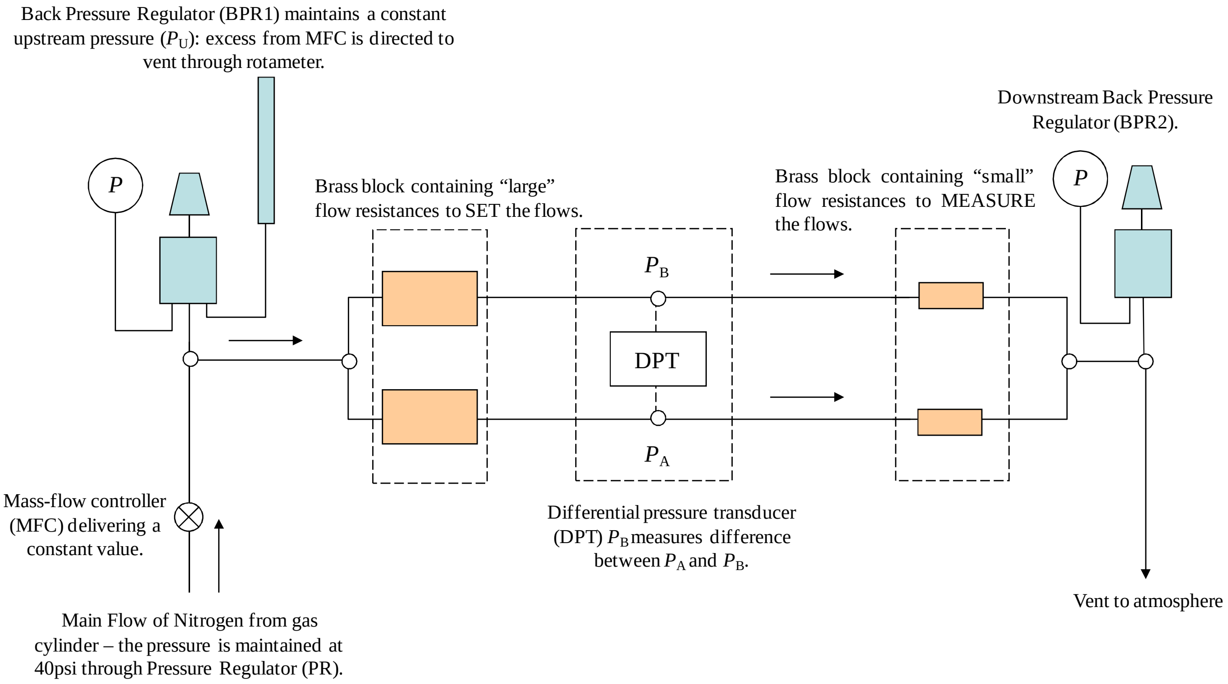

Figure 3.1 Experimental arrangement for baseline

## 3.3 Experimental arrangement for adding perturbation gas

The system described in Section 3.2 for the baseline is modified (Figure 3.2) to add small flow of nitrogen gas which has flowrate of around 1% of the main flowrate. This system is developed by making a connection from the main flow of nitrogen gas and then fitting a mass flow controller and discharge valve on the stream carrying this perturbation gas. When the valve is opened it likely that the DPT signal would change because of an increase in the $P_A$ and when the valve is closed the system would approach towards its baseline. This experimental setup is not the ultimate objective of the current research project, however it provides an understanding in terms of the expected response from adding ethanol vapours into the main flow of nitrogen gas.

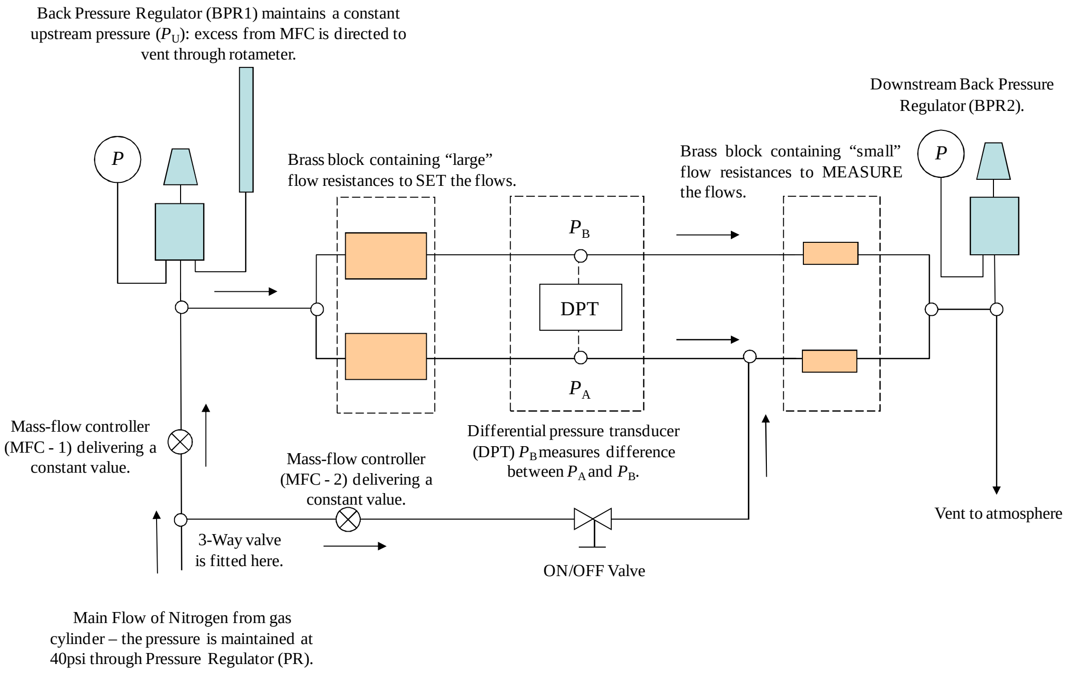

Figure 3.2 Adding small flow of perturbation gas into main stream

## 3.4 Integration of Microplant and diffusion cell

The experimental set-up described in Section 3.2 is integrated with the diffusion cell (Figure 3.3) to add a vapour flow of ethanol into the main flow of nitrogen gas. This is achieved with a T-piece fitting which connect the tubing carrying the main flow of nitrogen and the outlet diffusion tubing of the ethanol container. The container volume is 150ml which is filled 75% of its total volume with ethanol (95% pure), the ethanol vapours flows through a 12.7cm (5inch) diffusion tube having 3.175mm (1/8inch) diameter. At the middle of the diffusion tube there is on-off valve which could be opened to admit the vapours flow and closed to prevent the vapours flow.

The main purpose is to see a step change in the DPT signal when the valve is opened. When vapours flow through the diffusion tube and commingle with the main flow of nitrogen gas it results in an increase in $P_A$. There are many potential design parameters that are likely to affect the DPT response for example surface area of the ethanol container, distance from the liquid surface to the main flow of gas and volume of vapour space.

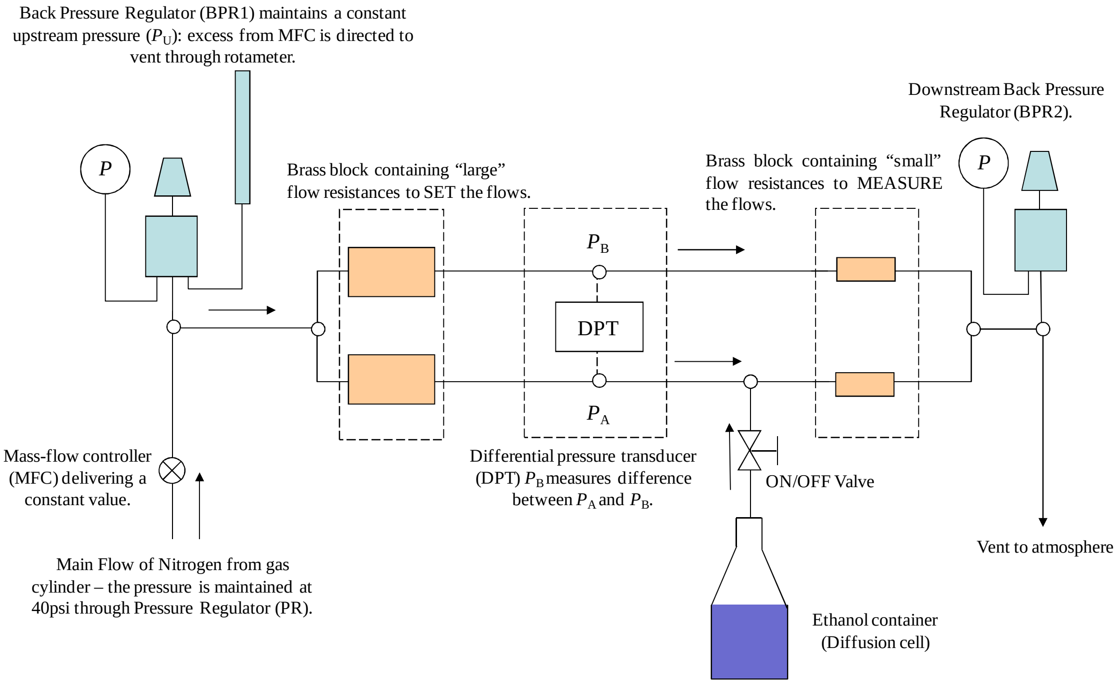

Figure 3.3 An integration of Microplant and diffusion cell

## 3.5 Modified arrangement of microplant and diffusion cell

The system described in Section 3.4 is further analysed and then modified to increase the flow of vapours from ethanol container. To achieve this ethanol container is placed in a water bath (Figure 3.4) the temperature of which is maintained at 70°C throughout the experiment. This system seems to provide a constant and an increased flow of ethanol vapours, however when the ethanol vapours leaves the container it is likely that vapours will condense when the temperature drops in the diffusion tube and/or in the nylon tubing. Therefore, this experimental arrangement is not followed for current study and the main focus is on the arrangement described in Section 3.4 which is carried out at room temperature and likely to give the required results of current research.

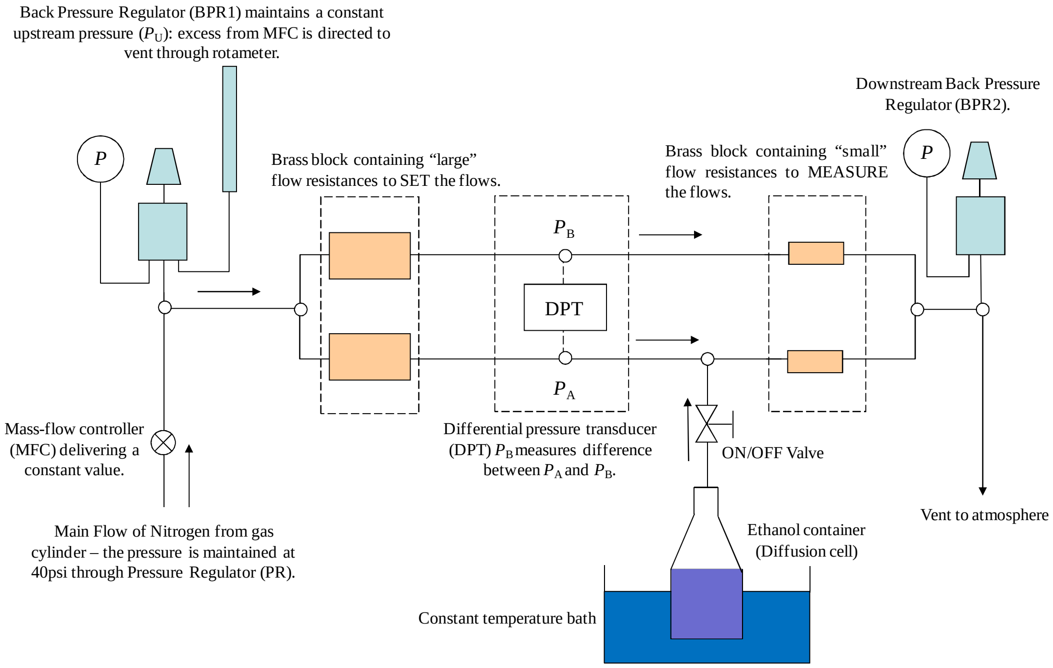

Figure 3.4 Modified system of integrated Microplant and diffusion cell

## 3.6 Determination of vapour concentration

The experimental system was first calibrated by adding nitrogen perturbation flow (~1% of the main nitrogen stream flowrate) into the main stream of nitrogen gas which resulted a step change in the DPT signal. This change in DPT signal was recorded and compared against the change in DPT signal caused by adding the ethanol vapours into the main stream of nitrogen gas.

The ethanol vapour concentration in the nitrogen gas stream is calculated as follows:

$$\text{Ethanol evaporation rate} = \frac{y}{x} \times [\text{Flowrate of Nitogen gas}]$$

Where,

*x* is the change in DPT signal due to adding small flow of nitrogen perturbation gas.

*y* is the change in DPT signal due to the addition of ethanol vapour to the nitrogen stream. .

# Chapter 4 Results and Discussion

## 4.1 Introduction

In this chapter the theoretical and experimental methods to determine the typical evaporation rate of ethanol into nitrogen stream are discussed. The results achieved from running different experiments on the experimental arrangements described in Chapter 3 for baseline, adding small flow of nitrogen gas into the main stream and adding ethanol vapours into the main stream of nitrogen gas are analysed and discussed.

## 4.2 Theoretical method

The typical evaporation rate of ethanol is calculated based on the theoretical method described in Section 2.2 of Chapter 2.

Diffusion tube length ($L$) = 5inch = 12.7cm

Diameter of diffusion tube ($d$) = 1/8inch = 0.3175cm

System Pressure ($P$) = 760mmHg = $101.3*10^3$Pa

System temperature ($T$) = 24°C

Cross sectional area of tube ($\Pi/4\ (d^2)$) = 0.0791 $\text{cm}^2$

Gas constant ($R$) = $8314.472*10^3$ $\text{Pa.cm}^3$/mol.K

Saturated vapour pressure of ethanol @24°C ($P_L$) = 43.5mmHg = 5798Pa

Diffusion Coefficient (Lee and Wilke, 1954) ($D$) = $0.135\text{cm}^2$/sec

Molecular weight of ethanol ($M$) = 46.07g/mol

Diffusion rate = $r$ =?

Diffusion rate equation:

$$r = \frac{DPMA}{RTL} \ln \pi \qquad 4.1$$

Where, $\pi$ = Pressure term ($P/(P-P_L)$)

By putting the value of pressure term Equation 4.1 becomes:

$$r = \frac{DPMA}{RTL} \ln\left( \frac{P}{(P - P_L)} \right) \qquad 4.2$$

Using the above date and Equation 4.2

$r = 9.36975*10^{-8}$g/sec

This is the evaporation rate of ethanol based on mass flow. The volumetric flowrate can be calculated as follows:

The ideal gas equation is given as:

$$PV = nRT \qquad 4.3$$

(Where $n = m/M$, and density is the mass per unit volume)

$$PM = \rho RT \text{ or } \rho = \frac{PM}{RT} \qquad 4.4$$

Based on the above data the density of ethanol vapour can be calculated as follows:

$$\frac{5798\text{Pa} \times (46.07\text{g/mol})}{(8314 \times 10^3\,\text{Pa.cm}^3\text{/mol.K}) \times (297.16\text{K})} = \text{is } 0.181 \times 10^{-3}\text{g/ml.}$$

$$\text{Volumetric flowrate} = \frac{\text{Mass flowrate}}{\text{Density}} \quad 4.5$$

Using Equation 4.5 the theoretical evaporation rate of ethanol is predicted to be 0.052ml/min.

## 4.3 Experimental method

In this section the results achieved from running a range of experiments on the MT alone and its integration with the diffusion cell are discussed and analysed.

### 4.3.1 Baseline

A number of experiments are run using the experimental arrangement for baseline as described in Chapter 3. The following Figure 4.1shows the result achieved for the baseline. It can be seen that in Figure 4.1 that all points are in the range of 0.02mV.

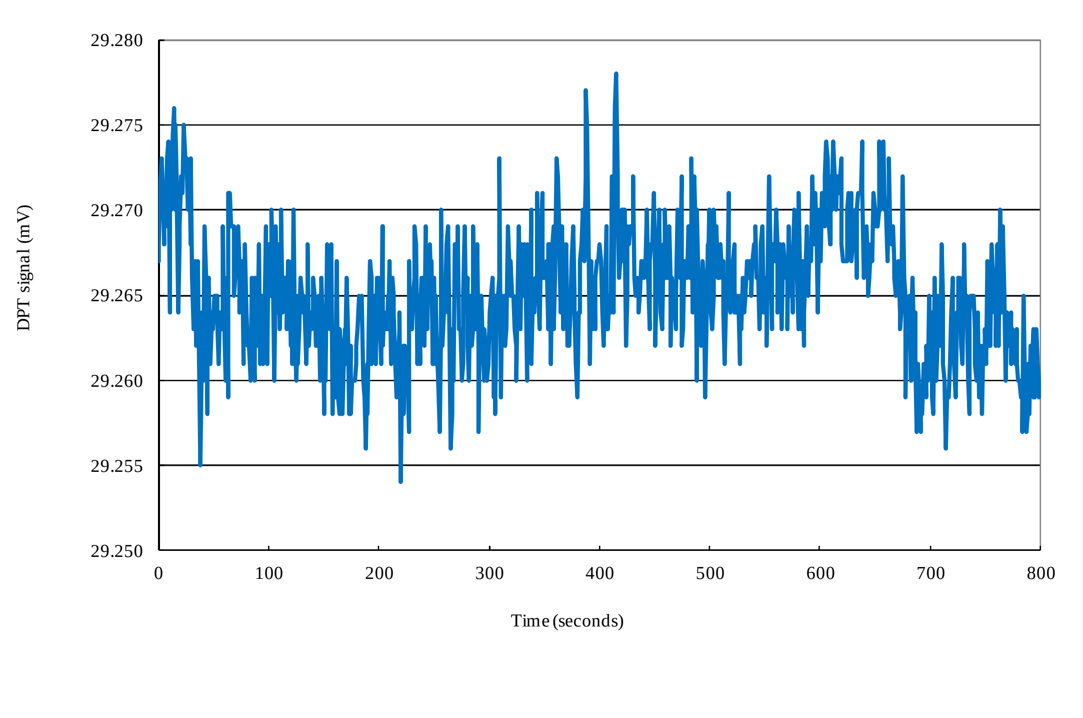

Figure 4.1 Standard baseline using DPT (±1000mm$H_2O$)

### 4.3.2 Adding a small flow of perturbation gas into main stream

As explained in Section 3.8 adding a small flow of perturbation gas would cause an increase in flowrate which as a result would increase the pressure.  Our study indicates that the perturbation flow is normally at flow rate of around 1% to that of the main flowrate. In the current experimental arrangement, adding a small flow of perturbation gas (nitrogen) into the main stream of nitrogen gas increases the pressure and this can be noticed as a step increase in the DPT signal.

In Figure 4.2 it can be seen very clearly that when the valve is opened there is a sudden step change (increase) in the DPT signal and the system approaches toward baseline when the valve is closed.. The spikes shown in the below Figure 4.2 are caused by the noise level that has been recorded by the DPT and can be minimised by reducing the noise level.

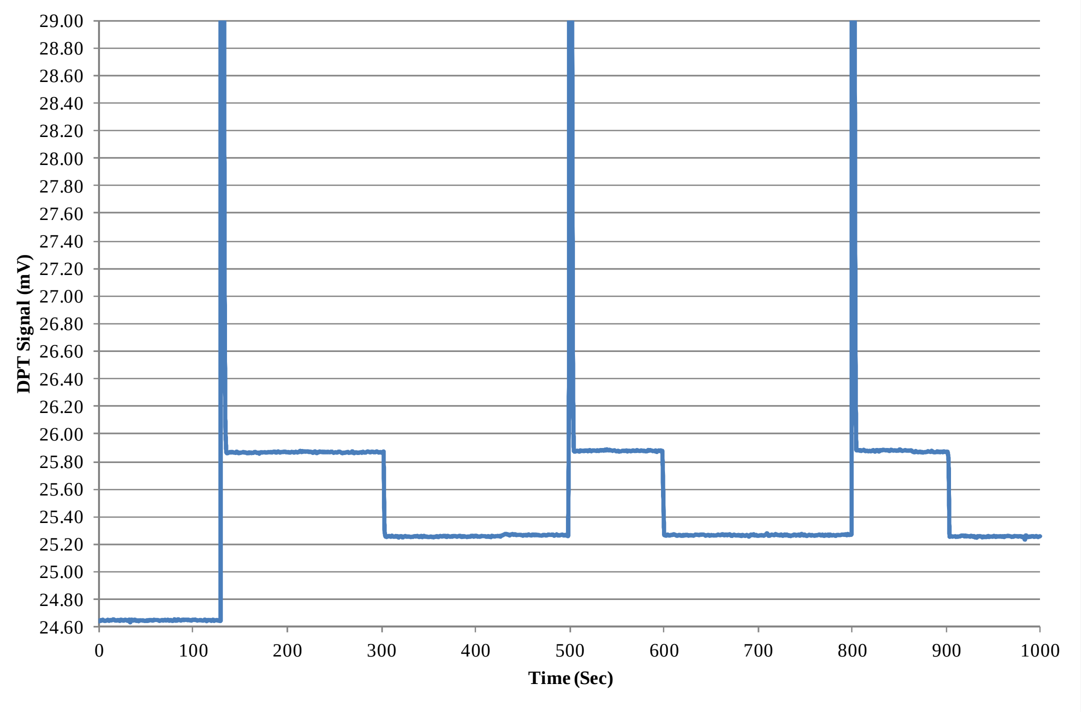

Figure 4.2 Adding perturbation flow (0.5ml/min) into main stream (52ml/min) - DPT (±1000mm$H_2O$)

### 4.3.3 Microplant and diffusion cell

In this section more or less the same response is expected as noticed in Section 4.3.2. The below Figure 4.3 shows a typical response when the valve is opened. In this experiment the valve is opened at around 40sec and then left opened until 400sec at which point the valve is closed. It can be seen that there is a sharp step change in the DPT signal as the valve is opened and the system then approaches towards it baseline. The noise band in this case is in the range of 0.5mV.

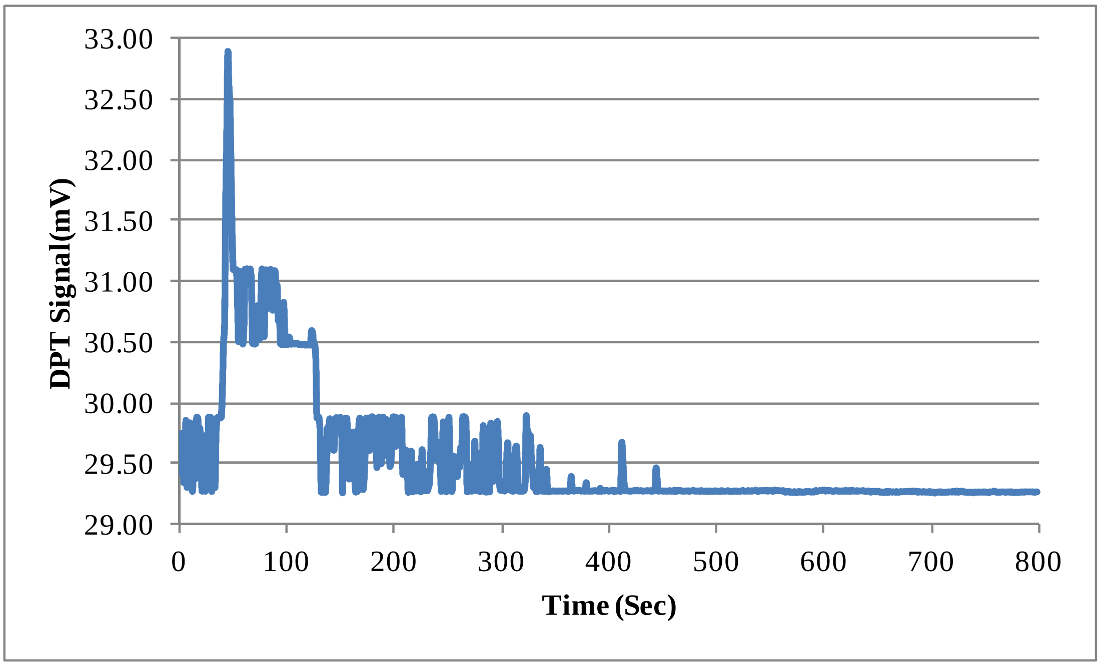

Figure 4.3 Effect of opening the valve on DPT Signal

### 4.3.4 Calculating experimental ethanol evaporation rate

To find out the rate of evaporation of ethanol when the valve is opened sensitivity analysis is carried out. In this analysis the recorded data of Figure 4.3 is analysed and compared with the results achieved from adding a nitrogen perturbation gas into the main stream of nitrogen gas in which case the flowrate of nitrogen perturbation gas is known (0.5ml/min). This comparison would help to determine the evaporation rate of ethanol from the current diffusion cell into the main stream of nitrogen gas.

First taking Figure 4.2 into consideration where the perturbation gas flow rate is 0.5ml/min. In this case when the valve is opened at ~130sec the DPT signal is increased from 24.64mV to 25.25mV. This indicates that when perturbation gas is added at a flowrate of 0.5ml/min the step change in the DPT signal increases by a value of 0.61mV.

Similarly, in Figure 4.3 in which case ethanol vapours are added into the main stream of nitrogen gas. In this case, when the valve is opened at 40sec the step change in DPT signal increases from 29.88mV to 29.90mV and the system then approaches towards it baseline value. This indicates that there is step change of 0.02mV in DPT signal which happened because of adding ethanol vapours into the main stream of nitrogen gas.

However, the ethanol evaporation rate is not known at this stage which resulted a step change of 0.02mV in the DPT signal. Therefore, this rate is calculated by making a comparison between the data of Figure 4.2 and Figure 4.3. This can be achieved through a simple mathematical equation as described in Section 3.6.

Ethanol evaporation rate = (0.5×0.02/0.61) = 0.0163ml/min

This value of 0.0163ml/min indicates the typical evaporation/diffusion rate of ethanol from the current diffusion cell.

### 4.3.4 Comparison of theoretical and experimental results

Analysing the results achieved from both theoretical and experimental methods it can be seen that the evaporation rate by theoretical method is 3.2 times greater than the evaporation rate through experimental method. However, this discrepancy can be reduced by refining the data and also applying minimum design changes to the current diffusion cell more specifically changing the dimensions of diffusion tube i.e. increasing the diameter of diffusion tube and also reducing the length from liquid surface to the main stream of nitrogen gas.

# Chapter 5 Conclusions and Future Work

## 5.1 Conclusions

A vaporiser (diffusion cell) is designed and developed that gives a flow of vapours (ethanol) into a gas stream (nitrogen) at normal temperature and atmospheric pressure. This diffusion cell is then integrated with microplant technology to measure the evaporation rate through change in flowrate. The evaporation rate of vapours (ethanol) into nitrogen gas stream is also predicted through theoretical method and compared against the results achieved from experimental method.

In terms of experimental arrangement, a microplant is developed and a number of experiments are run for the baseline which indicated a noise band of around 0.02mV (0.2Pa) using Furness Control DPT (±1000mm$H_2O$). The microplant is then modified to add a small flow of perturbation gas (nitrogen) into the main stream of nitrogen gas. This is just to see and make sure that there is a step change in DPT signal due to increase in pressure when a small flow of perturbation gas (nitrogen) is added into the main stream of nitrogen gas. To determine the evaporation rate of ethanol the microplant is then integrated with the diffusion cell in which case ethanol vapours from the diffusion cell are added into the main stream of nitrogen gas.

The results indicated that when the valve restricting the perturbation gas (nitrogen) is opened and a small flow of perturbation gas (0.5ml/min) is added into the main stream of nitrogen gas (52ml/min) then there is a step change in the DPT signal i.e. the pressure increases when the perturbation gas is added. However, when the valve is closed there is sharp decrease in the DPT signal and the system then approaches towards the baseline.

Similarly, when ethanol vapours are added into the main stream of nitrogen gas the step change of 0.02mV was noticed in the DPT signal. Based on the step changes in DPT signal from adding perturbation gas and adding ethanol vapours the calculated ethanol evaporation rate is 0.0163ml/min.

In light of the results achieved from theoretical method it was found that the evaporation rate of ethanol is 0.052ml/min. However, from experimental method the diffusion rate of ethanol into the main stream of nitrogen gas is only 0.0163ml/min. Comparing both results the evaporation rate predicted through theoretical method is 3.2 times greater than the evaporation rate by experimental method. This discrepancy is large and can be reduced by applying design changes to the current diffusion tube i.e. increasing its diameter and reducing the distance from the liquid surface and the main stream of nitrogen gas.

It should be noted that in the current experiments there was noise impact even when the valve was closed. This noise impact was increased when the valve was opened to add the ethanol/perturbation gas to the main stream of nitrogen gas. This noisy data makes the reproducibility of the experiments difficult. Based on this the valve closure experiments are preferred because it avoids the pressure spikes.

## 5.2 Future work and recommendations

In the current work diffusion tube having dimension of 1/8inch (3.175mm) diameter and length 5inch are used, however in future a larger diameter should be selected which can allow a smooth flow of ethanol vapours into the main stream of nitrogen gas.

Secondly, the distance between the liquid surface and the main stream of nitrogen gas should be reduced which could be achieved by reducing the length of the diffusion tube. The ethanol container used in the current work isn’t uniform in dimension, however in future a container should be selected which is uniform in dimension and also provides sufficient surface area at which point there is possibility of more evaporation from the liquid surface.

In the current integration of microplant and diffusion cell a suitable adsorbent should be fitted downstream which would remove the moisture content in the gas stream and then the remaining moisture content would be measured using the chemical engineering concepts of flowrate measurements and material balances. This would give an alternative method of moisture measurement at a very low level.

In the current work there is a noise impact and because of that noisy data is recorded which in most of the cases misleading an observer. There should be an option in the Pico software which only records the actual data of the experiment and ignores the noisy data.  Filtering the data would be an option, however this option only considers a range of data rather than taking the actual experimental data into consideration and ignoring the noisy data. In future, an effort should be made to further minimise the impact of noisy data.

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# 7 Appendix

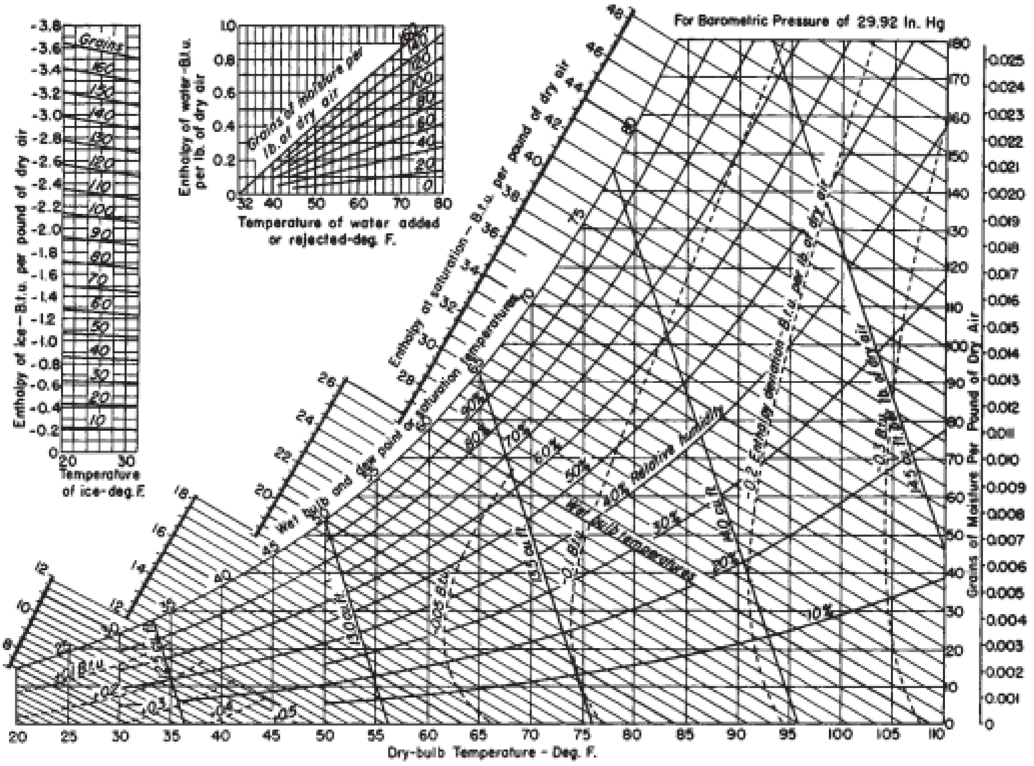

Figure 7.1 Psychrometric Chart—medium temperatures, barometric pressure 29.92inHg.