Department of Chemical Engineering

Unit Operation Lab

ChE-422 Manual

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STUDENT NUMBER:

SEMESTER / YEAR:

EXPERIMENT #01 (UO-01)

MOLECULAR DIFFUSION IN GASES

OBJECTIVE:

To determine the diffusion coefficient

THEORITICAL BACKGROUND

Molecular diffusion or molecular transport can be defined as the transfer or movement of individual molecules through a fluid by means of the random, individual movements of the molecules.

The diffusivity of the vapor of a volatile liquid in air can be determined by winklemann's method in which liquid is contained in a narrow diameter vertical tube, maintained at a constant temperature. A stream is passed over the top of the tube to ensure that the partial pressure of the vapor is transferred from the surface of the liquid to the air stream by molecular diffusion. With this method, the diffusion is the same as gas A diffusing in stagnant, nondiffusing B.

$$N_A = D\left(\frac{C_A}{L}\right)\left(\frac{C_T}{C_{Bm}}\right) \tag{2.1}$$

The evaporation of the liquid A is expressed as follows

$$N_A = \frac{\rho_L}{M}\frac{dL}{dt} \tag{2.2}$$

Equating the above equations, integrating and rearranging the resulting equation, one can obtain as follows:

$$\frac{t}{(L-L_0)} = \left(\frac{\rho_L}{2MD}\right)\left(\frac{C_{Bm}}{C_A C_T}\right)(L-L_0) + \left(\frac{\rho_L C_{Bm}}{MDC_A C_T}\right)L_0 \tag{2.3}$$

The above equation is indeed a linear correlation between $\frac{t}{(L-L_0)}$ against $(L-L_0)$ with slope = $\left(\frac{\rho_L}{2MD}\right)\left(\frac{C_{Bm}}{C_A C_T}\right)$

Thus by plotting $\frac{t}{(L-L_0)}$ versus $(L-L_0)$, one can easily calculate the slope and find the diffusivity coefficient of gas A in B.

$$C_T = \frac{1}{22.414}\left(\frac{T_{Abs}}{T_a}\right) \tag{2.4}$$

$$C_{Bm} = \frac{(C_{B1}-C_{B2})}{ln\left(\frac{C_{B1}}{C_{B2}}\right)} \tag{2.5}$$

$$C_{B1} = C_T \tag{2.6}$$

$$C_{B2} = \left(\frac{P_a - P_V}{P_a}\right) C_T \tag{2.7}$$

$$C_A = \left(\frac{P_V}{P_a}\right) C_T \tag{2.8}$$
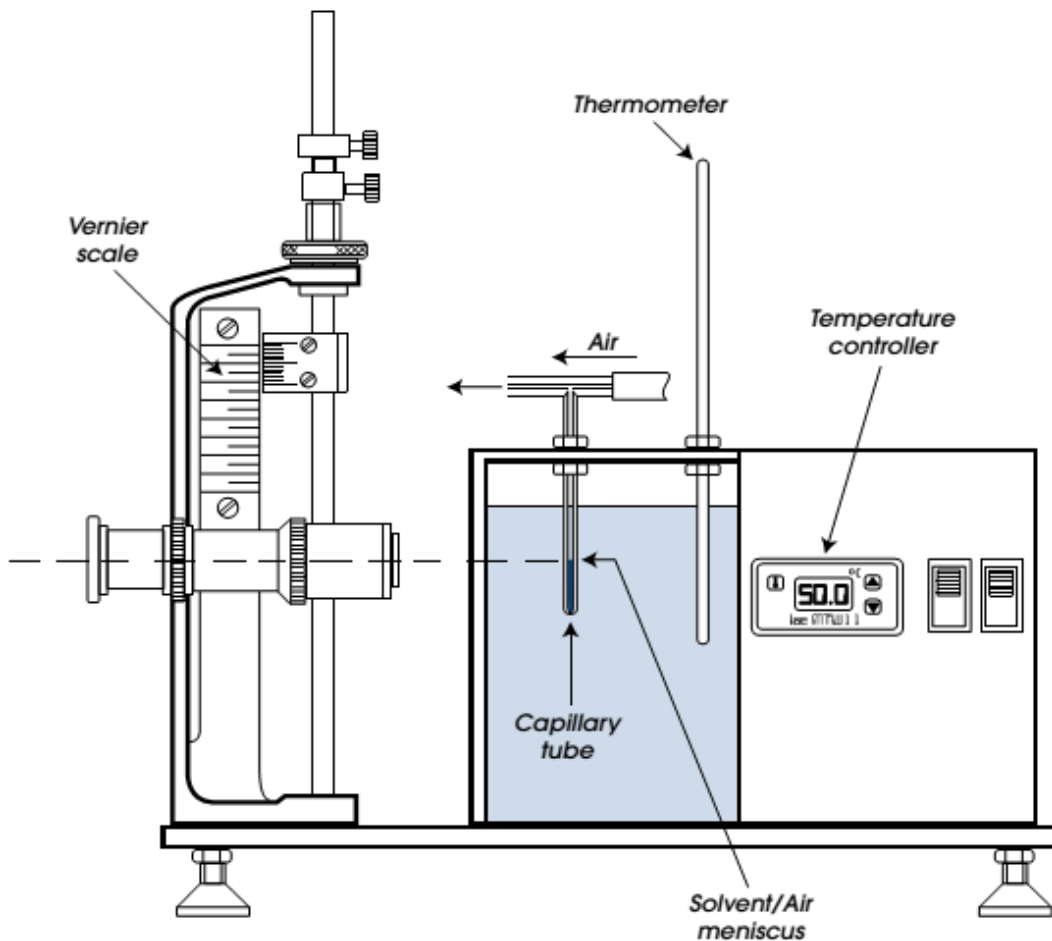
DESCRIPTION OF THE EQUIPMENT

Figure 2.1. Linear Conduction Heat Transfer Unit

The liquid that is to be volatilized is placed in capillary tube. The capillary tube is placed inside a water bath whose temperature is carefully controlled by a controller. The height of the liquid that decreases by time is observed using microscope equipped with Vernier height gauge.

EXPERIMENTAL PROCEDURE

1. Fill the capillary tube with acetone to a depth of approximately 35 mm.
2. Insert capillary tube through a rubber ring, inside the metal nut until the top of tube rest on the top of the nut.

3. Screw gently the above assembly onto the top plate, with the 'T' piece normal to the microscope
4. Connect flexible air tube to one end of the 'T' piece.
5. Set up the microscope as shown in figure 4.1. and adjust the object lens to within 20-30 mm from the tank.
6. Adjust the vertical height of the microscope until the capillary tube is visible (if the capillary tube is still not visible adjust the distance from the object lens to the tank until it is visible).
7. Adjust viewing microscope until the meniscus inside the capillary tube clear and well defined (note that the image in the microscope is upside down. That means the top of the image is the bottom of the capillary tube).
8. Make sure that the sliding Vernier scale should be aligned with a suitable graduation on the fixed scale.
9. Switch on the air pump.
10. Adjust the air flow rate using the Hoffman clip on the flexible tube so the flow rate is low.
11. Record the level inside the capillary tube.
12. Switch on the temperature controlled water bath and adjust set point on controller to 40 °C to obtain steady temperature at 40 °C.
13. Switch off the temperature controlled water bath after approximately 60 minutes to prevent air bubbles.
14. Record the change in level inside the capillary tube.
15. Repeat #12 - #14.

REFERENCES

Geankoplis,C.J., "Separation Processes and Unit Operations, 4th edition, Prentice-Hall Inc., New Jersey, 2004

"CERa Experiment Instruction", armfield, 2011**.**

NOTATION

C_A	:	Saturation concentration of acetone at interface, kmol/m^3
C_{Bm}	:	Logarithmic mean molecular concentration of vapor, kmol/m^3
C_T	:	Total concentration, kmol/m^3
D	:	Diffusivity coefficient, m^2/s
L	:	Effective distance of mass transfer (m)
M	:	Molecular weight of acetone, kg/mol
N_A	:	Molar flux, mol/m^2.sec
P_a	:	Atmospheric pressure, atm
P_V	:	Vapor pressure of acetone, atm
ρ_L	:	Density of acetone, kg/m^3
T	:	Time, sec
T_a	:	Bath temperature, K
T_{abs}	:	Absolute temperature, K

WORK SHEET

Time, ks	Liquid level $(L\text{-}L_0)$	$\dfrac{t}{(L-L_0)}$

Do the following:

1. Plot between $\dfrac{t}{(L-L_0)}$ and $(L-L_0)$
2. Calculate diffusivity coefficient from the slope of the line.
3. Compare your findings with **theoretical value**. (refer to mass transfer course)

EXPERIMENT #02 (UO-02)

CONVECTION DRYING

OBJECTIVE:

1. To determine the reduction in weight and the drying rate over time during the drying process.
2. To investigate the effect of air flow rate in convection drying.

THEORITICAL BACKGROUND

Drying is a physical process of removing the liquid adhering to a material and contained within it. In drying process, the water is usually removed as a vapor by air. It proceeds more slowly for the moisture that is located in capillaries and pores within the material to be dried. This is because the capillary forces or diffusion resistance must be overcome. The removal of water of crystallization contained in the material to be dried is even more difficult because it is bounded by relatively strong molecular forces. The material must be intensely heated to expel the water.

According to the type of heat transfer, drying process can also be classified as follows:

1. Convection drying when the heat for the drying is transferred to the material to be dried by a flowing gas.
2. Contact drying when the heat is transferred from the heater to the material by conduction due to physical contact between them.
3. Radiation drying when the heat is transferred by radiation. The source of energy can be in form of electromagnetic radiation, infrared, microwave or even radio frequency.

In many cases, drying is carried out using warm air at atmospheric pressure. This drying process is called air drying. If drying is done at significant sub atmospheric pressure, it is called vacuum drying. In some cases, the moisture in the material is cooled and frozen and using convection, conduction or radiation heat transfer, and the freezing moisture can be sublimed. This method of drying is known as freezing drying.

One of the most important analyses in drying is rate of drying curves. The equilibrium moisture contents of various materials cannot be predicted and must be determined experimentally. Similarly, since the basic mechanism of rates of drying is quite incomplete thus it is necessary in most cases to obtain some experimental measurements of drying rates.

DESCRIPTION OF THE EQUIPMENT
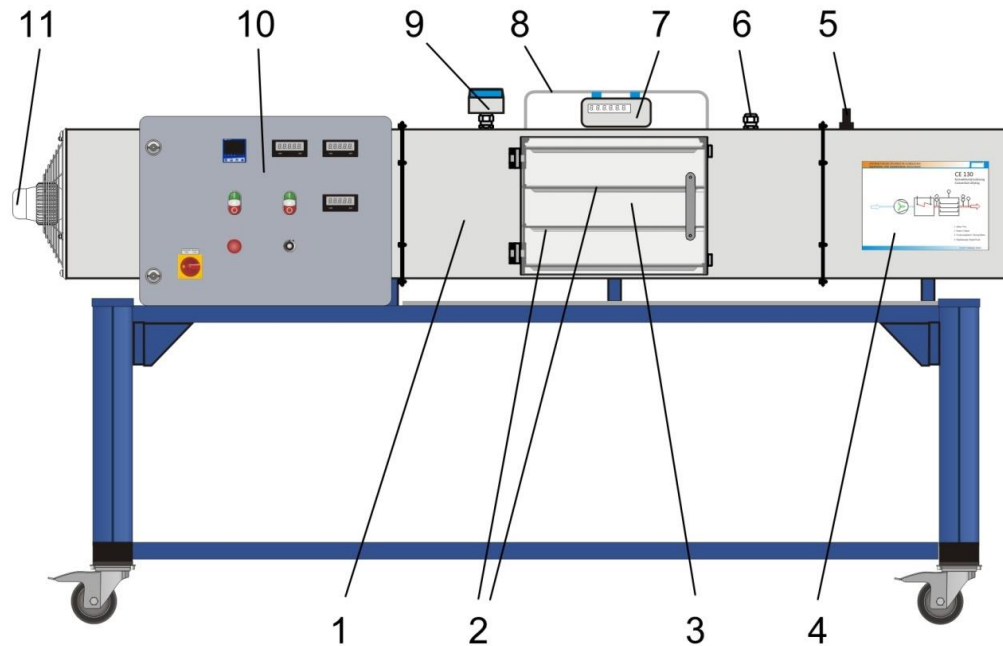
1. Drying channel
2. Drying plates
3. Transparent door
4. Process schematic
5. Air velocity sensor
6. Measuring point for humidity and temperature
7. Digital balance
8. Bracket for fying plates
9. Measuring point with humidity and temperature sensor
10. Switch cabinet with digital displays
11. Fan

EXPERIMENTAL PROCEDURE

1. Remove drying plates from support frame.
2. Tare scale to zero
3. Insert drying plates individually into support frame, read off and note the weight of the individual drying plates on the digital scale.
4. Switch on fan and set speed using flow rate gauge e.g. 1.3 m/s.
5. Read off volumetric air flow from display and make a note of it.
6. Switch on heater and set the temperature controller e.g. 50°C.
7. Run air conditioning duct until it is warmed up evenly all along its length.
8. Spread material (gravel with grain size of 0.5-0.7 mm) to be dried in a thin layer on the drying plates.
9. Insert drying plates in the support frame and note the total weight.
10. Tare scale to zero.

11. Introduce water to the spread material e.g. 50 g
12. Start drying experiment, commence time measurement using stopwatch.
13. Record air temperature T_2 after material to be dried, relative humidity after material being dried, total mass m of material to be dried (material and drying plates).
14. Stop the experiment when the mass m of material to be dried does not change significantly.
15. Repeat the experiment for air speed of 2 m/s.

REFERENCES

Transport Processes and Separation Process Principles, 4th ed., 2003, C.J. Geankoplis, Pearson Education, Inc.

CE 130 GUNT Experiment Manual.

WORK SHEETS

Experiment #1

Mass of drying plate 1   :

Mass of drying plate 2   :

Mass of drying plate 3   :

Dry Gravel mass   :

Introduced water   :

Total Mass   :

Air speed   :

Heating level   :

Time min	T_1 °C	φ_1 % R.H.	T_2 °C	φ_2 % R.H.	W_T g
	Before plate		After plate		

Experiment #2

Mass of drying plate 1   :
Mass of drying plate 2   :
Mass of drying plate 3   :
Dry Gravel mass   :
Introduced water   :
Total Mass   :
Air speed   :
Heating level   :

Time min	T_1 °C	φ_1 % R.H.	T_2 °C	φ_2 % R.H.	W_T g
	Before plate		After plate		

1. Plot the moisture content versus time for both experiments, comment on it in the discussion part.
2. Plot the drying rate versus time for both experiments, determine which part is constant drying rate period and which part is falling rate period. comment on it in the discussion part.
3. Find the graphical value of constant drying rate.
4. predict value of constant drying rate.
5. Compare the value from #3 and #4. Comment on the result.

EXPERIMENT #03 (UO-03)

WET COOLING TOWER

OBJECTIVE:

To investigate and understand the influence of volumetric air flow rate, volumetric water flow rate and packing density on the performance parameters of the cooling tower.

THEORITICAL BACKGROUND

Water or wet cooling tower is one of the most important types of process in the process industries. This type of cooling tower follows the principle of evaporative cooling. There are many cases in industry in which warm water is discharged from heat exchangers and condensers when it would be more economical to cool and reuse it than to discard it.

In a typical water-cooling tower, warm water flows counter currently to an air stream. Typically, the warm water enters the top of a packed tower and cascades down through the packing, leaving at the bottom. Air enters at the bottom of the tower and flows upward through the descending water. The tower packing often consists of slats of plastic or packed bed. The water is distributed by troughs and overflows to cascade over slat gratings or packing that provides large interfacial areas of contact between the water and air in the form of droplets and films of water.

In this cooling tower, water evaporates into air because its vapor pressure in the air is less than the saturation vapor pressure. The amount of heat that is needed to evaporate the liquid is drawn from the air. Furthermore, this air cools water in contact with it. In this case, the wet-bulb temperature which takes both temperature and humidity into account, as compared to the actual air temperature (dry-bulb temperature), is a measure of the potential for evaporative cooling. The greater the difference between the two temperatures, the greater the evaporative cooling effect. Therefore, wet-bulb temperature is essentially the lowest temperature which can be attained by evaporative cooling at a given temperature and humidity.

Several characteristic variables are calculated as follows

Cooling range which is the difference between the warm water temperature at the inlet to the cooling tower and the cold water temperature at the outlet from the cooling tower

$$z = T_4 - T_5 \tag{1.1}$$

Wet bulb approach which is the difference between the cold water temperature at the cooling tower outlet and the cooling limit or wet bulb temperature.

$$a = T_5 - T_{f1} \tag{1.2}$$

Cooling coefficient is formulated as follows

$$\eta = \frac{T_4 - T_5}{T_4 - T_{f1}} \tag{1.3}$$

Water loss due open circuit in cooling tower is calculated as follows

$$\dot{m}_W = (X_2 - X_1)\dot{m}_L \tag{1.4}$$

Heat load which is the heat which is taken from the water during the cooling process is formulated

$$\dot{Q}_W = \dot{m}_W C_{pW} z \tag{1.5}$$

Actual air mass flow rate is calculated as follows:

$$\dot{m}_L = \alpha \varepsilon c \sqrt{\frac{(P_1 - P_2)}{v_2}} \tag{1.6}$$

α = 0.605; ε = 0.98, c = 0.0073 and the volumetric air flow is calculated

$$\dot{V}_L = \dot{m}_L v_2 \tag{1.7}$$

Cooling capacity which is the difference in enthalpy between the air inlet and outlet

$$\dot{Q}_L = (h_2 - h_1)\dot{m}_L \tag{1.8}$$

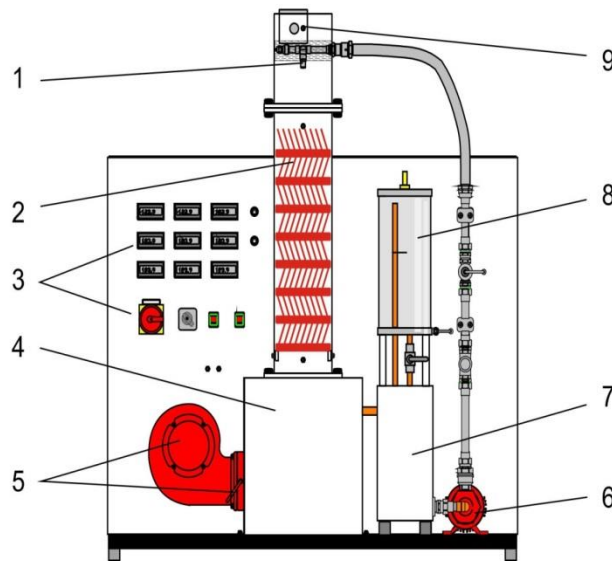**DESCRIPTION OF THE EQUIPMENT**

Figure 1.1 Wet Cooling Tower

The main wet cooling tower unit can be seen in figure 1. The water is heated using heating element in a tank (7), pumped (6) and then sprayed from the top of the column through nozzle (1) and intimately contact with cooling air due to the packing (2). The air comes in from the air chamber (4) at the bottom of the column. The air flow rate is adjustable using fan (5). Due to

evaporation during the course of the process, the make-up water (8) is required to maintain enough water for the process. All parameter readings including water and air flow rate as well as temperature can be collected manually from the display and control buttons (3). The column is also equipped with temperature/humidity sensor (9) at the top and the bottom.

EXPERIMENTAL PROCEDURE

Prior to experiment, it is important to do the following steps:

Experiment #1: investigation on the effect of water flow rate.

1. Fill the water tank and the supply tank (make-up water tank)
2. Choose the packing type 1 and assemble it.
3. Connect all sensors (combined temperature/humidity and temperature sensors)
4. Connect the system to the mains electricity supply.
5. Turn on the heater and select the heating level 2 i.e. 1 kW and heat the water in the tank up to 40 °C.
6. Turn on the fan and adjust the air flow rate so the pressure difference is approximately 120 Pa by changing the shutter opening.
7. Turn on the pump and adjust the volumetric water flow rate at 60 L/h.
8. After reaching steady state (app. 20 mins), take all measurement readings from the display and fill out worksheet.
9. Repeat the same procedure for water flow rate of 75 L/h and 90 L/h.

Experiment #2: investigation on the effect of air flow rate.

1. Reduce the speed down to 60 L/h.
2. After reaching steady state (app. 10 mins), take all measurement readings.
3. Repeat the same procedure for Δp of 140 pa and 160 Pa.

Experiment #3: investigation on the effect of packing density.

1. Turn off the heater, fan and pump. Disconnect all sensors and replace the packing type with type #4.
2. Connect all sensors (combined temperature/humidity and temperature sensors)
3. Connect the system to the mains electricity supply.
4. Turn on the heater and select the heating level 2 i.e. 1 kW and heat the water in the tank up to 40 °C.
5. Turn on the fan and adjust the air flow rate so the pressure difference is approximately 120 Pa by changing the shutter opening.
6. Turn on the pump and adjust the volumetric water flow rate at 60 L/h.
7. After reaching steady state (app. 20 mins), take all measurement readings from the display and fill out worksheet.

REFERENCES

Geankoplis,C.J., "Separation Processes and Unit Operations, 4th edition, Prentice-Hall Inc., New Jersey, 2004

Jebavy,F. ,"WL 320 Experiment Instruction", GUNT, 2007.

NOTATION

a	:	Wet bulb approach, K
c	:	Constant, m^2
C_{pW}	:	Specific heat capacity of water, kJ/kg.K
h_1	:	Enthalphy of outlet air, kJ/kg
h_2	:	Enthalphy of inlet air, kJ/kg
m_L	:	Air mass flow rate, kg/s
m_W	:	Water mass flow rate, kg/s
P_1	:	Inlet pressure, Pa
P_2	:	Outlet pressure, Pa
Q_W	:	Heat load, W
T_4	:	Inlet water temperature, K
T_5	:	Outlet water temperature, K
T_{f1}	:	Wet bulb temperature, K
V_L	:	Air volumetric flow rate, l/s
X_1	:	Absolute humidity at outlet air, g/kg
X_2	:	Absolute humidity at inlet air, g/kg
z	:	Cooling range, K
α	:	Flow Coefficient
ε	:	Expansion Coefficient
η	:	Cooling Coefficient
ν_2	:	Kinematic Viscocity, m^3/kg

WORK SHEETS

Experiment #1

Parameter	Run 1	Run 2	Run 3
A_B, m^2/m^3	110	110	110
T_1, °C			
φ_1, %			
T_2, °C			
φ_2, %			
T_4, °C			
T_5, °C			
ΔP, Pa	120	120	120
F_w, l/min	60	75	90

Experiment #2

Parameter	Run 1	Run 2
A_B, m^2/m^3	110	110
T_1, °C		
φ_1, %		
T_2, °C		
φ_2, %		
T_4, °C		
T_5, °C		
ΔP, Pa	140	160
F_w, l/min	60	60

Experiment #3

Parameter	Run 1
A_B, m^2/m^3	200
T_1, °C	
φ_1, %	
T_2, °C	
φ_2, %	
T_4, °C	
T_5, °C	
ΔP, Pa	120
F_w, l/min	60

.EXPERIMENT #04 (UO-04)

FLUIDIZED BED

OBJECTIVE:

1. To investigate the loosening speed of a fluidized bed in air and water medium.
2. To determine the relationship between flow rate and the depth of the fluidized bed.
3. To investigate the effect of particle size on fluidization.

THEORITICAL BACKGROUND

Fluidized beds are widely used in process technology. One of important applications in chemical technology is fluidized bed reactor in which variety of multiphase chemical reactions is carried out. In a fluidized bed, a layer of fine granular solid matter is loosened by a fluid flowing through it to such extent that the particles of solid matter are free to move within certain limits. The layer of solid material takes on similar properties to a fluid.

To characterize a fluidized bed, the pressure loss of the fluid flowing through the bed can be used. When a fluid flows through the mass initially the pressure underneath the mass increases as the flow speed increases until the pressure forces match the weight of the mass, and the mass becomes suspended. With further increasing flow rate, the layer set in motion and reaches a fluidized state. At this condition the static solid-like state changes to dynamic-like state. The pressure loss now remains almost constant even with further increasing flow rate. From a certain flow rate the particles at the top no longer fall back into the fluidized bed, they are drawn off by the fluid flow and removed.

Pressure losses in fluidized bed can be calculated as follows:

$$\Delta p = g \left(1 - \frac{\rho_f}{\rho_p} \right) h \rho_{ps} \tag{3.1}$$

Meanwhile loosening speed at which the mass of solid matter passes the transition to a fluidized bed is expressed

$$w_{lo} = \frac{Re_{lo}}{d_p} v_f \tag{3.2}$$

For irregular shape, loosening speed must be corrected,

$$w = w_{lo} \varphi \tag{3.3}$$

The void fraction of a bed is expressed
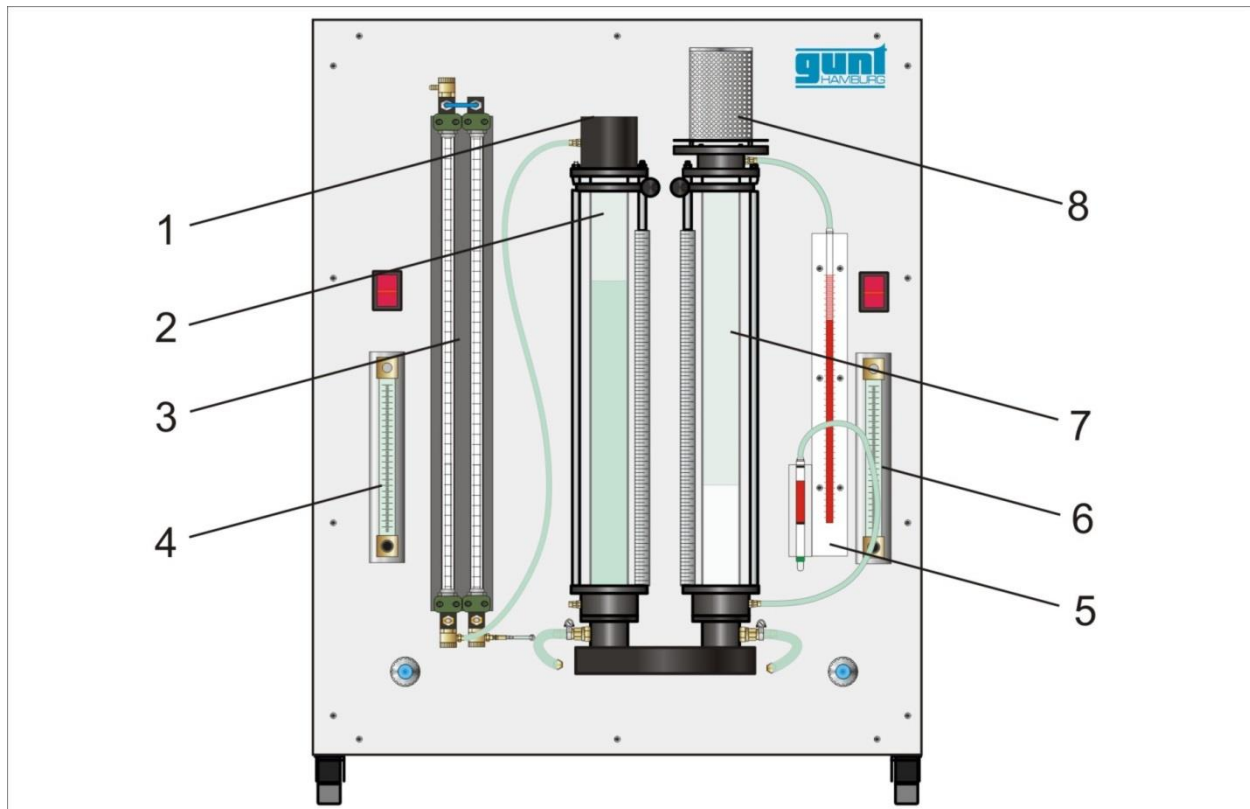
$$\varepsilon = 1 - \frac{\rho_{ps}}{\rho_p} \tag{3.4}$$

Reynolds' number is formulated as follows

$$Re_{lo} = 42.86(1 - \varepsilon)\left(\sqrt{1 + 3.11 \times 10^{-4} A_r \frac{\varepsilon^3}{(1-\varepsilon)^2}} - 1\right)$$ (3.5)

With Archimedes' number is defined as follows

$$A_r = \frac{g d_p^3}{\mu^2} \frac{\rho_p - \rho_f}{\rho_f}$$ (3.6)

1. water overflow 2. test tank for water 3. 2-tube manometer 4. flow meter for water 5. U-tube manometer 6. flow meter for air 7. test tank for air 8. filter

EXPERIMENTAL PROCEDURE

Filling, emptying, and cleaning the single tube manometer, 2 tube manometer, and vessel with particles, including the calibration and testing of vessel for both water and air must be done by the trained person.

A. Calibration with empty test tank for water and air
1. Connect the pressure measuring connections to the manometer. On a two tube manometer, the display value can be set to the center of the manometer.
2. Fully open the by pass valve below the rotameter
3. Fully close the needle valve on the rotameter.
4. Turn on the pump

5. Increase the flow in small increments by opening the needle valve
6. Continuously note the flow rate and differential pressure in the table.
7. Continue the measurements up to the maximum flow.

B. Experiment with test tank for water
1. Record the initial bed height (h=100 mm) and ensure particle with d_p of 0.505 mm is used.
2. Fully open the bypass valve below the rotameter.
3. Fully close the needle valve on the rotameter.
4. Turn on the pump.
5. Increase the flow in small increments by opening the needle valve.
6. Observe the particle movement, note the flow rate, bed depth, and pressure drop.
7. Continue increase the flow rate until flow rate of 1.5 L/min is obtained (above certain flow rate, the needle valve will be already fully open, further increase of flow rate must be done by closing the bypass valve).

C. Experiment with test tank for air
1. Fill the test vessel with particle i.e. d_p = 0.240 mm, until the height of the bed is 50 mm.
2. Fully open the by pass valve below the rotameter.
3. Fully close the needle valve on the rotameter.
4. Turn on the compressor.
5. Increase the flow in small increments by opening the needle valve and observe the mass.
6. Continuously take note of the flow rate and differential pressure.
7. Take the note as soon as the first signs of the particle movements.
8. Continue until the a flow 30 L/min is achieved (above certain flow rate the needle valve will be already fully open, further increase of flow rate must be done by closing the bypass valve)
9. Repeat the same procedure for different particle size, i.e. d_p=0.505 mm.

REFERENCES

"CE220 Experiment Instruction", GUNT, 2011**.**

NOTATION

ΔP	:	Pressure drop, mm
G	:	Gravitational acceleration, m/s^2
ρ_f	:	Fluid density, kg/m^3
ρ_p	:	Particle density, kg/m^3
H	:	Height of mass
ρ_{ps}	:	Particle mass density, kg/m^3
w_{lo}	:	Fluid velocity between spherical particles, m/s
Re_{lo}	:	Reynolds' number
d_p	:	Particle diameter, m

v_f	:	Kinematic viscocity
W	:	Corrected fluid velocity, m/s
φ	:	Form factor
ε	:	Void fraction
Ar	:	Archimedes' number
η	:	viscocity

WORK SHEET

A. Calibration with empty test vessel for water and air.

For water

Q, L/min	ΔP,mm	W, m/s

For Air

Q, L/min	ΔP,mm	W, m/s

B. Experiment with test vessel for water.

Dp =   mm

Q, L/min	ΔP,mm	H, mm	W, m/s

C. Experiment with test vessel for air.

D_p = mm

Q, L/min	ΔP,mm	W, m/s

D_p = mm

Q, L/min	ΔP,mm	W, m/s

Do the following:
1. Plot pressure drop against fluid speed for all data.
2. Plot bed depth against flow rate for test vessel with water only.
3. Determine the loosening velocity (W_{lo}) for experiment B and C from the plot.
4. Determine the loosening velocity (W_{lo}) using equation and compare with that obtained from the plot.
5. Comment on the all above plots.

EXPERIMENT #06 (UO-06)

SOLID LIQUID EXTRACTION

OBJECTIVE:

1. To study the performance of discontinuous solid-liquid extraction process.
2. To study the influence of number of stages on counter-flow solid-liquid extraction process.
3. To study the effect of solvent temperature on the extraction performance.

THEORITICAL BACKGROUND

Many biological, inorganic, and organic substances occur in a mixture of different components in solid. Solid-liquid extraction is general term used for a process to separate either desired or undesired solute from the solid phase using a solvent/liquid. It is sometimes called *leaching* and if it is intended to remove undesirable component from solid using water, it is called *washing*.

In general the process of solid-liquid extraction can be described as follows; the solvent must be transferred from the bulk solvent solution to the surface of the solid. The solvent then must penetrate or diffuse into the solid and the solute dissolves into the solvent. The solute then diffuses through the solid solvent mixture to the surface of the particle. Finally, the solute is transferred to the bulk solution.

There are several factors influencing extraction performance for example:

1. Differences in concentration; the greater the difference in concentration of the usable substance in the extraction material and in the solvent, the greater the driving force when extracting.
2. Surface area; it is proportional to the extracted quantity of material.
3. Diffusion resistance; it depends on the size of the particles, the porosity and the penetrability of the extraction material for the solvent. The diffusion resistance should be as low as possible.
4. Temperature; The higher the temperature, the better the performance of the extraction.

Extraction can be done at different pattern as follows

1. Cross flow; the extraction material is sprayed with fresh solvent at each stage.
2. Uni-flow; the extraction material is mixed with solvent and move continuously from stage to stage in uni-flow pattern.
3. Counter flow; extraction material and solvent move continuously in a counter flow.

The counter flow method generally represents the optimum option. Compared to the uniflow method, a considerably higher proportion of usable substance is transferred from the extraction material into solvent. The concentration gradient as the driving force for the mass transfer is utilized more effectively with the counter flow method. Cross flow method results in even higher yields but requires a much more fresh solvent.

DESCRIPTION OF THE EQUIPMENT

Figure 5.1. Solid-Liquid Extraction Unit

Solid-liquid extraction unit as shown in the diagram (1) has a spiral conveyor (2) to feed the extraction material into revolving extractor in which the extraction process occurs. This spiral conveyor can feed the solid up to 20 l/h with power consumption up to 4 W. Revolving extractor is driven by a motor (4) with speed up to 9 h^{-1} and power consumption up to 0.9 W. The solvent is then fed using several peristaltic pumps (5) into the revolving extractor. The pump can feed the solvent at maximum volumetric flow rate of 25 l/h at 300 rpm. Solvent (liquid), extract (liquid) and raffinate (solid residue) are charged into the tanks (6). The mode of extraction can be changed by changing the selector valves (7) and the solvent can also be heated using a heater (8) with power consumption of 330 W. All reading and controls are placed in a cabinet (9).

EXPERIMENTAL PROCEDURE

Preparation of Extraction Material

1. Add 10 L distilled water and 600 g potassium hydrogen carbonate and mix them until the salt is completely dissolved.
2. Add approximately 4 kg of aluminium oxide grains in the above solution, make sure all grains are perfectly immersed in solution and keep it in solution for 24 hours.
3. Take out the grains from the solution and dry it (if needed baking tray or oven can be used).

Experiment #1 : Extraction Performance for discontinuous process

1. Set the valve position V_1 and V_2 to 1 stage which means the solvent is pumped to the first stage.
2. Weigh 70 g or aluminium oxide grains and add it to the cell extractor.
3. Make sure the cell extractor i.e. first stage is exactly positioned below the solvent feed.
4. Turn on process pump P_1 and set the solvent flow rate of 15 l/h and turn on heating element W_1 and set the temperature of 30 ^{o}C.
5. Take the concentration reading at specific time interval (as can be seen in worksheet).

Experiment #2: Effect of solvent temperature on extraction performance

1. Make sure the filling hopper is filled with sufficient quantity of solid material.
2. Synchronize the speed of the solid feeder and the extractor so that a material depth approximately 40 mm per cell is achieved.
3. Set the valve position V_1 and V_2 to 1 stage which means the solvent is pumped to the first stage.
4. Turn on process pump P_1 and set the solvent flow rate of 15 l/h and turn on heating element W_1 and set the temperature of 25 ^{o}C.
5. Take the concentration reading at specific time interval e.g. every 30 secs (as can be seen in worksheet).
6. Increase the temperature to 30 ^{o}C once the extract concentration from previous experiment does not increase anymore.
7. Take the concentration reading at specific time interval i.e. every 30 secs.
8. Increase the temperature to 40 ^{o}C once the extract concentration from previous experiment does not increase anymore.
9. Take the concentration reading at specific time interval i.e. every 30 secs.

Experiment #3: Effect of solvent flow-rate on extraction performance

1. Make sure the filling hopper is filled with sufficient quantity of solid material.
2. Synchronize the speed of the solid feeder and the extractor so that a material depth approximately 40 mm per cell is achieved.
3. Set the valve position V_1 and V_2 to 1 stage which means the solvent is pumped to the first stage.
4. Turn on process pump P_1 and set the solvent flow rate of 10 l/h and turn on heating element W_1 and set the temperature of 30 ^{o}C.
5. Take the concentration reading at specific time interval i.e. every 30 secs (as can be seen in worksheet).

6. Increase the flow rate to 15 l/h once the extract concentration from previous experiment does not increase anymore.
7. Take the concentration reading at specific time interval i.e. every 30 secs.
8. Increase the flow rate to 20 l/h once the extract concentration from previous experiment does not increase anymore.
9. Take the concentration reading at specific time interval i.e. every 30 secs.

Experiment #4: Influence of number of stages on extraction performance (optional)

1. Make sure the filling hopper is filled with sufficient quantity of solid material.
2. Synchronize the speed of the solid feeder and the extractor so that a material depth approximately 40 mm per cell is achieved.
3. Set the valve position V_1 and V_2 to 1 stage which means the solvent is pumped to the first stage.
4. Turn on process pump P_1 and set the solvent flow rate of 15.5 l/h and turn on heating element W_1 and set the temperature of 30 °C.
5. Turn on process pump P_2 and P_3. Adjust the speed in such a way that as few air bubbles as possible occur in the relevant intake line.
6. Take the concentration reading at specific time interval i.e. every 30 secs (as can be seen in worksheet).
7. Set the valve position V_1 and V_2 to 2 stage which means the solvent is pumped to the first and second stage.
8. Keep the setting on process pump P_1 the same as step #4.
9. Turn on and adjust the speed of process pump P_2 and turn on heating element W_2 and set the temperature of 30 °C.
10. Take the concentration reading at specific time interval i.e. every 30 secs (as can be seen in worksheet).
11. Set the valve position V_1 and V_2 to 3 stage which means the solvent is pumped to the first, second and third stage.
12. Keep the setting on process pump P_1 the same as step #4 and that on process pump P_2 as step #9.
13. Turn on and adjust the speed of process pump P_3 and turn on heating element W_3 and set the temperature of 30 °C.
14. Take the concentration reading at specific time interval i.e. every 30 secs (as can be seen in worksheet).

REFERENCES

Geankoplis,C.J., "Separation Processes and Unit Operations, 4th edition, Prentice-Hall Inc., New Jersey, 2004

"CE 630 Solid Liquid Extraction", GUNT, 2012.

WORK SHEETS

Experiment #1 : Extraction Performance for discontinuous process

Solvent flow rate　　　:　　　l/h

Solvent temperature　:　　　ºC

Time,s	Conc., g/l	Time,s	Conc., g/l	Time, s	Conc., g/l	Time, s	Conc.,g/l
0		150		360		630	
15		180		390		750	
30		210		420		870	
45		240		450		1000	
60		270		480		1360	
90		300		510		1720	
120		330		570		2000	

Experiment #2 : Effect of solvent temperature on extraction performance

Solvent flow rate　　　:　　　l/h

*indicate when you start to change the temperature.

Time,s	Cond-4	Time,s	Cond-4	Time, s	Cond-4	Time, s	Cond-4
0		1170		2340			
30		1200		2370			
60		1230		2400			
90		1260		2430			
120		1290		2460			
150		1320		2490			
180		1350		2520			
210		1380		2550			
240		1410		2580			
270		1440		2610			
300		1470		2640			
330		1500		2670			
360		1530		2700			
390		1560		2730			
420		1590		2760			
450		1620		2790			
480		1650		2820			
510		1680		2850			
540		1710		2880			
570		1740		2910			
600		1770		2940			
630		1800		2970			
660		1830		3000			
690		1860		3030			

720		1890		3060			
750		1920		3090			
780		1950		3120			
810		1980		3150			
840		2010		3180			
870		2040		3210			
900		2070		3240			
930		2100		3270			
960		2130					
990		2160					
1020		2190					
1050		2220					
1080		2250					
1110		2280					
1140		2310					

Experiment #3 : Effect of solvent flow rate on extraction performance

Solvent Temperature :   °C

*indicate when you start to change the flow rate

Time,s	Cond-4	Time,s	Cond-4	Time, s	Cond-4	Time, s	Cond-4
0		1170		2340			
30		1200		2370			
60		1230		2400			
90		1260		2430			
120		1290		2460			
150		1320		2490			
180		1350		2520			
210		1380		2550			
240		1410		2580			
270		1440		2610			
300		1470		2640			
330		1500		2670			
360		1530		2700			
390		1560		2730			
420		1590		2760			
450		1620		2790			
480		1650		2820			
510		1680		2850			
540		1710		2880			
570		1740		2910			
600		1770		2940			
630		1800		2970			

660		1830		3000						
690		1860		3030						
720		1890		3060						
750		1920		3090						
780		1950		3120						
810		1980		3150						
840		2010		3180						
870		2040		3210						
900		2070		3240						
930		2100		3270						
960		2130								
990		2160								
1020		2190								
1050		2220								
1080		2250								
1110		2280								
1140		2310								

Experiment #4 : Effect of number of stage on extraction performance

Solvent Temperature : ^{o}C

*indicate when you start to change the flow rate

Time, s	Cond -2	Cond -3	Cond -4	Time, s	Cond -2	Cond -3	Cond -4	Time , s	Cond -2	Cond -3	Cond -4
0				1170				2340			
30				1200				2370			
60				1230				2400			
90				1260				2430			
120				1290				2460			
150				1320				2490			
180				1350				2520			
210				1380				2550			
240				1410				2580			
270				1440				2610			
300				1470				2640			
330				1500				2670			
360				1530				2700			
390				1560				2730			
420				1590				2760			
450				1620				2790			
480				1650				2820			
510				1680				2850			
540				1710				2880			

570				1740				2910			
600				1770				2940			
630				1800				2970			
660				1830				3000			
690				1860				3030			
720				1890				3060			
750				1920				3090			
780				1950				3120			
810				1980				3150			
840				2010				3180			
870				2040				3210			
900				2070				3240			
930				2100				3270			
960				2130							
990				2160							
1020				2190							
1050				2220							
1080				2250							
1110				2280							
1140				2310							

Student tasks:

1. Plot concentration (yE, g/L) against time (t, sec) for every experiment. Separate the plot if there is change in parameter such as temperature or flow rate. Comment on the plots.
2. Do material balance for the solid-liquid leaching once the system reach steady state (experiment #2 and #3)

EXPERIMENT #07 (UO-07)

PARTICLE SIZE ANALYSIS (SIEVING) and ANGLE OF REPOSE

OBJECTIVE:

1. To determine and analyze the size distribution of a fixed granular solid
2. To determine the natural angle of repose for a specific materials.

THEORITICAL BACKGROUND

Small particle is one of chemical engineering interest. An understanding of the characteristics of masses of particulate solids is necessary in designing processes and equipment for dealing with streams containing such solids.

One way to distinguish the particle size is by sieving. In each test sieve consists of a woven wire screen with square apertures of known size and is used for the measurement of particle size. The necessary vibrating motion is imparted by a mechanical shaker to ensure reproducible results in a relatively short time.

Particle size may be specified by quoting the size of two screens, one through which the particles have passed and the other on which they are retained. However sieving is usually used to measure size distribution. The results of sieve test may be presented in a variety of ways either in tabular or graphical form.

The other way to learn characteristics of solid material is by measuring the angle of repose. The angle of repose is the steepest possible angle with the horizontal at which the material will stand when piled. Moisture content of the material is often a controlling factor and the percentage of fine material in the mass has a decided influence on the angle as the fine materials carry the bulk of moisture. Similarly, the angle depends upon the type of material including the shape and smoothness of the individual particles and their overall compactness.

DESCRIPTION OF THE EQUIPMENT

EXPERIMENTAL PROCEDURE

A. Sieving Experiment

1. Weigh material e.g. sand that will be separated or graded using shaker and sieves. Use the last container which is the one at the bottom of sieve series to make sure that the volume of material does not exceed the volume of sieves.
2. Transfer the material to another container or beaker.
3. Put the sieve with the biggest screen size at the top and the smallest at the bottom (see fig 5.1)
4. Set shaker time to 20 min to make sure the material is completely graded.
5. Carefully detach each sieve and weigh the collected material from each sieve.
6. Record the mass obtained from each sieve in the work sheet.
7. Repeat the experiment twice.

B. Angle of Repose in a Hele-Shaw Cell

1. Place test material (dry or wet) in the funnel.
2. Measure the angle of repose by sliding the cell and repeat.
3. Empty the Hele-Shaw Cell by rinsing with water if required.
4. Ensure dry berfore refilling the system with another material
5. Repeat the experiment 2 times for each type of material.

REFERENCES

Instruction Manual for CEN-MKII, Armfield.

WORK SHEETS

Run #1

Screen Opening, mm	Average particle diameter	Mass, g
	Total mass	

Run #2

Screen Opening, mm	Average particle diameter	Mass, g
	Total mass	

Run #3

Screen Opening, mm	Average particle diameter	Mass, g
	Total mass	

Run	Material Tested and its condition	Angle of Repose
1		
	Average	
2		
	Average	
3		
	Average	
1		
	Average	
2		
	Average	
3		
	Average	

EXPERIMENT #08 (UO-08)

SEDIMENTATION

OBJECTIVE:

1. To study sedimentation process and investigate the zone occurs in sedimentation tank.
2. To study the effect of flow rate of fresh water and suspension in sedimentation process.

THEORITICAL BACKGROUND

Sedimentation is a physical process of separating suspended solid in a liquid using gravity. The suspended solids can settle down if the size is greater than 10 μm. the smaller the size the more difficult for the solids to settle down because Brownian motion and electrostatic force balances the gravitational force. Unless coagulant is added, it is not likely to settle down very small particle naturally.
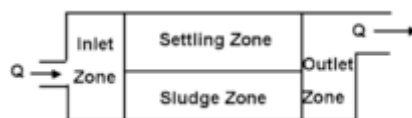
Figure 6.1. The Four Functional Zone of Continuous Sedimentation Process

In typical sedimentation tank, there will be four functional zones. Inlet zone is the zone where all solid particles are well mixed and flow in the same direction as the liquid. The settling occurs in settling zone as the liquid continue flowing to outlet zone. Most of solid particles will be settled and collected continuously in the sludge zone.

In this experiment, to determine the actual sediment content at inlet and outlet to the sedimentation tank, samples of the mixtures are taken. This batch system will show four different zones as can be seen in figure 6.2. In hindered settling, particles become quite close and the liquid is flowing upward due to displacement by settling particles. This will reduce the particle settling velocity.
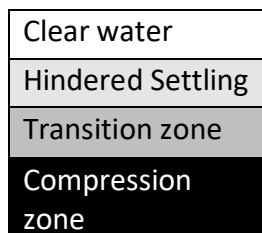

Figure 6.2. The four zones of Batch Sedimentation

At the bottom of sedimentation column, the particles can contact each other and indeed the particle concentration becomes very high. The settled solids are compressed by the weight of overlying solids and water is squeezed out while the spaces between solid particles are getting smaller. This zone is known as compression zone. Between hindered settling zone and compression zone there will be transition zone.

DESCRIPTION OF THE EQUIPMENT
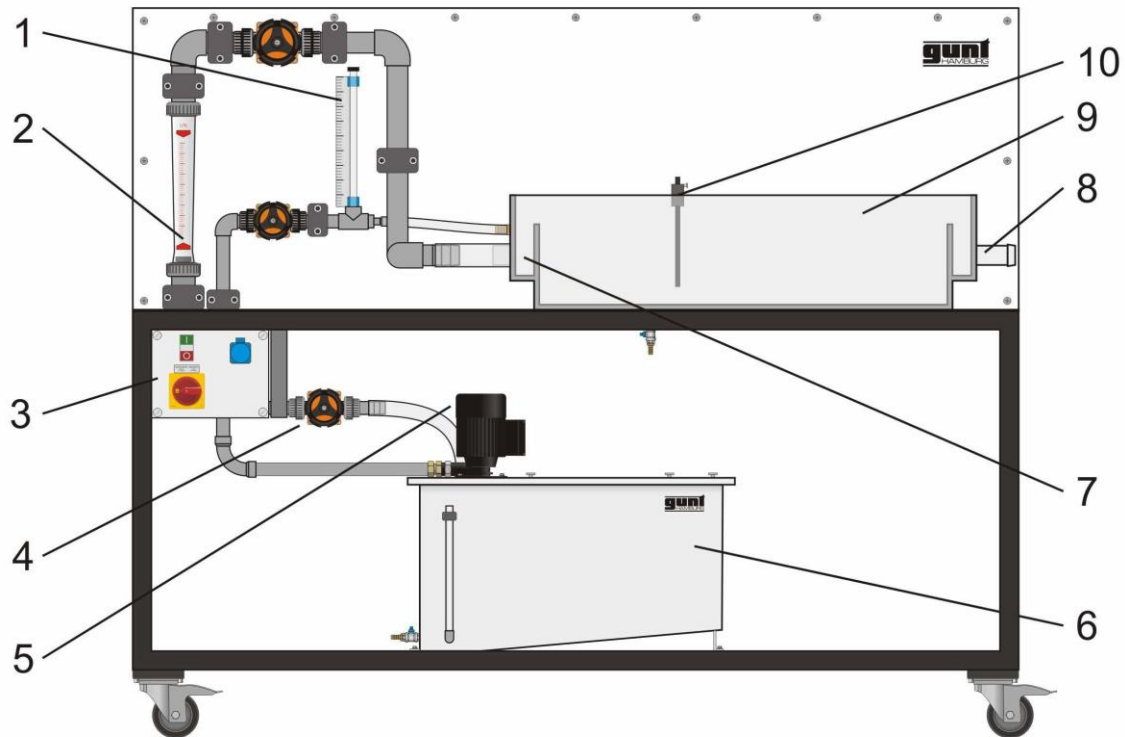
1. Suspension flow meter
2. Fresh water flow meter
3. Switch box
4. Bypass
5. Suspension pump
6. Suspension tank
7. Fresh water/suspension mixing zone
8. Outlet
9. Sedimentation tank
10. Baffle plate

Figure 6.3. Sedimentation Unit

EXPERIMENTAL PROCEDURE

Preparation

1. Close fresh water, suspension and by pass valves.
2. Close drain valve of suspension tank.
3. Ensure that no hose lies in the suspension tank.
4. Fill the suspension tank with 80 L of fresh water, then add 1.6 kg of precipitated calcium carbonate.
5. Fully open bypass valve.
6. Switch on main switch and pump switch. The suspension is now mixed by the pump.

Performing the experiment

1. Drain sedimentation tank via drain valve and rinse with fresh water.

2. Remove or insert baffle plate (depending upon the mode of operation: undisturbed or disturbed sedimentation. If disturbed sedimentation is chosen then insert the baffle approximately 400 mm from inlet weir. Set a gap of 20-30 mm between bottom of baffle plate and base of sedimentation tank).
3. Insert plug into the inlet weir.
4. Close drain valve of sedimentation tank.
5. Carefully open fresh water valve and fill the sedimentation tank with fresh water, then set desired flow rate.
6. Move the scale on the riser for the suspension flow meter such that the zero mark is aligned with the water level in the riser.
7. Carefully open suspension valve and set desired flow rate (the suspension mixes with the fresh water in the inlet chamber and flows over the inlet weir into the sedimentation tank.
8. Keep checking suspension flow meter for constant flow rate, adjust suspension valve if necessary.
9. After 10 minutes, open the valves at both sampling points at the inlet and let the suspension flow to sedimentation cone.
10. Collect 1000 mL of the suspension from the inlet.
11. Immediately afterwards, open the valve at the sampling point at the outlet and let the suspension flow to another sedimentation cone.
12. Collect 1000 mL of the suspension from the outlet as well.
13. After 24 hour settling duration, read the amount of sediment in the sedimentation cones and compare (you can use flashlight to shine the sedimentation cone to enhance reading).

REFERENCES

HM 142 Gunt Experiment Manual.

NOTATION

WORK SHEETS

Time	Fresh water flow rate in l/h	Suspension flow rate in l/min	Amount of sediment in sedimentation cone at inlet in ml	Amount of sediment in sedimentation cone at outlet in ml	Calculated amount of sediment in sedimentation tank in ml
	100	0.5			
	100	1			
	100	2			
	200	2			
	400	2			

EXPERIMENT-9

Gas Absorption

Outcomes /Learning Objectives

- To investigate the absorption process when separating gas mixtures in a packed column
- Determination of pressure losses in the column
- To investigate the variables influencing the effectiveness of Gas absorption process

9.1 INTRODUCTION AND THEORITICAL EXPERIMENT

Gas absorption is a unit activity where dissolvable parts of a gas blend are broken up in a fluid. Most gas absorption goes for division of acidic pollutions from blended gas streams. These acidic polluting influences incorporate carbon dioxide (CO_2), hydrogen sulfide (H_2S), sulfur dioxide (SO_2), and natural sulfur mixes. The most significant of these are CO_2 and H_2S, which happen at convergence of five to fifty percent. Gas ingestion at a modern scale is most normally polished in pressed towers. A pressed pinnacle is basically a bit of pipe set on its end and loaded up with dormant material or "tower pressing." Liquid filled the highest point of the pinnacle streams down through the pressing; gas siphoned into the base of the pinnacle streams counter right now upward. The private contact among gas and fluid accomplished along these lines impacts the gas ingestion. Absorption is a basic procedure in thermal process engineering. Absorption is used to separate one or more gas components from a gas flow using a solvent (detergent). The CE 400 Gas Absorption unit provides a clear method of separating a gas mixture containing air and CO_2 by absorption in water.

In the gas mixture section, air is mixed with CO_2. Mixing is carried out manually using 2 control valves and 2 rotameters. In the absorption section, the CO_2 is washed out of the gas mixture in a glass absorption column using water as solvent. In the desorption section, the CO_2 is separated from the water in a glass desorption column. The key component of the trainer is the glass packed column, in which the gas mixture is brought into contact with water. During operation, samples of the gas mixture are taken at the gas inlet, in the center of the column and at the gas outlet.

9.2 Formulae(s) used

Calculation of air volumetric flowrate under normal conditions according to the thermal state equation for ideal gases:

$$Vair, st.\ Cond = Vair, Exp \times \frac{P_{Exp}T_{st.Cond}}{P_{st.cond}T_{Exp}} \tag{9.1}$$

Calculation of air molar flowrate is given by $Nair = \frac{Vair,st.CondVm}{V_m}$ (9.2)

Where V_m is the molar volume = 22.4 ltr/mol

Calculation of Water flowrate: $Naq = \frac{V_{aq}\, \rho_{aq}}{M_{aq}}$ (9.3)

Where $\rho_{aq} = 999.869$ g/litre and $M_{aq} = 18.02$ g/mol

Determination of loading:

The gas loading inlets is defined as

$$Y_E = \frac{N_{CO2.E}}{Nair}$$ (9.4)

$$Y_A = \frac{N_{CO2.A}}{Nair}$$ (9.5)

$$N_{CO2.E} = \frac{\varphi_E}{1 - \frac{100/\varphi_E}{100}}$$ (9.6)

$$N_{CO2.A} = \frac{\varphi_A}{1 - \frac{100/\varphi_A}{100}}$$ (9.7)

Where φ_E and φ_A *are the* inlet and outlet concentrations respectively

The CO_2 loading difference in the gas flow between inlet and the outlet is calculated

as $\Delta Y = Y_E - Y_A$ (9.8)

Description of the Equipment

Equipment: The GUNT CE 400 gas absorption unit will be used for the absorption of carbon dioxide in water. The process diagram of the CE 400 gas absorption unit is shown in Figure 1.
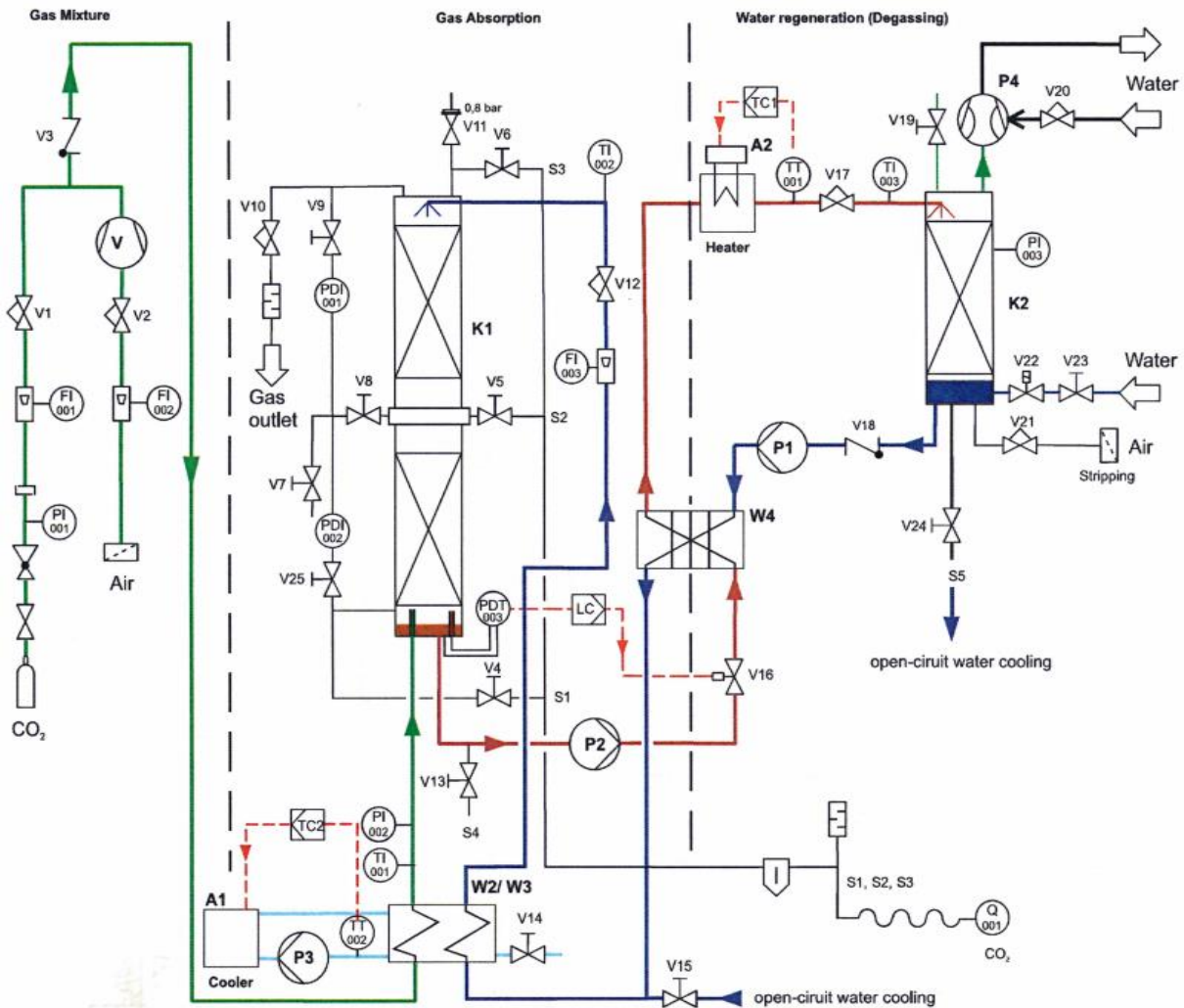
Figure 1 Process diagram for the gas absorption system

Standard operating procedure CE400 Gas Absorption

Procedure: Spend about 10 to 15 minutes to find out what are the physical parts that correspond to the components and measuring point on the process diagram.

Starting up the system

1. Turn on the gas absorption unit using the master switch.
2. **Refrigeration circuit filling.** Close V14 and fill up the tank W2/W3 with water as far as the inspection glass.
3. **Turn on the ready-to-use cooler, and set cooler temperature to 12 °C.** The cooler should be turned on first so that the water in tank W2/W3 can be cooled down to setpoint before the introduction of CO_2.
4. **Fill the U-tubes.** Close ball valves V9, V8 and V25; then open the 3 ball valves at the top of two U-tube manometers. Add water up to the ZERO line using a wash bottle in the outer vent holes. Then close the 3 ball valves above the U-tube manometers.
5. **Fill the system with absorbent (water)**. Close valves V13, V24, V15, V8, V25, V6, V5, V4, V21, V20 and open valves V10, V19, V23, V17, V12. Then press and hold the "Water supply" button on the controller cabinet until K2 is half full with water.

6. **Turn on pumps P1 and P2.** Water is pumped to the absorption column by P1, and recycled to the regeneration column by P2. Set the water flow rate to around 200 L/h using valve V12. If the level controller works correctly, a water level of 5 to 10 cm will be established above the base of the column. If the water level in the absorber cannot be maintained, turn off pump P1 and P2 and deaerate the pressure-measuring tubes with the screws opposite the connectors and keep the tubes free of air bubbles, and then turn on the pumps P1 and P2 again.

7. Set pressure in the regeneration column K2. To do this, close valve V19, open control valve V20 and set pressure to around 0.5 bar. If pressure in K2 falls below this, slightly open the control valve V21 and let "stripping" air into the regeneration column from the bottom of K2.

8. Open valves V9, V8 and V25.

9. Open valve V10, and close V2.

10. Turn on the compressor and slowly open valve V2 to adjust the air flow rate to 5 L/min.

11. Adjust the opening of V10 so that a higher than atmospheric pressure can be maintained in the absorber and adjust V2 accordingly to maintain a flowrate of 5 L/min.

12. When the water flow and gas flow become stable, record the pressure drop in the absorber using the U-tube manometer.

	PD1 (mmH_2O)	PD2 (mmH_2O)
300 L/h water, 5 L/min air		
300 L/h water, 30 L/min air		

13. Change the air flow rate to 30L/min and record the pressure drop.

14. After recording the pressure drop, slowly close valve V2 and then close ball valves V8, V9 and V25.

15. Reopen valve V2 and adjust the air flow rate to 5 L/min.

16. Set the pressure in K2 to 0.2 bar.

17. **Connect the CO_2 gas supply.** The set outlet pressure on the cylinder pressure reducer should be 0.8 bar.

18. **Introduce CO_2 into the system.** Adjust the control valve V1 to set the CO_2 gas volumetric flow rate to 1.2 L/min.

19. When the system comes to stable conditions, note down the following readings.

	temperature (°C)	pressure (bar)	flow rate (L/min)
Ambient conditions			N/A
Compressed air			
CO_2			
water		N/A	

20. **Measure the CO_2 concentration at three different point of the column K1: bottom S1, centre S2, and top S3.** Open the valve for the selected gas sampling point and allow the gas mixture to escape. Wait until it has displaced the ambient air from the water separator and the connection hoses (around 15-20 seconds). Then carry out the CO_2 measurement at the sampling point after the water separator using the CO_2 gas analyser, and take measurement over a period of 30 minutes at 5 minutes intervals. **NOTE: Before the first measurement, you should ensure that a gas cycle took place in the column.**

	Series of measurements (vol.%)							Mean values
	1	2	3	4	5	6	7	$\bar{X}$
	Time (hh:mm)							
S1								
S2								
S3								

Turning off the system

1. Shut down the CO_2 gas volumetric flow.
2. Turn off the cooler.
3. Disconnect the vacuum water jet pump and simultaneously open the valve V19 to equalise the pressure.
4. Turn off the compressor.
5. Turn off both pumps P1 and P2.

EXPERIMENT PROCEDURE

1. Start up the system. – Set the operating point – Turn on the cooling unit (adjust the set point temperature at 20°C).

2. To cool the system down more quickly, the cooler should be turned on around 1/2h

3. beforehand. Heater: on (adjust the set point temperature at 40°C).

4. Set the Water flow rate 300 ltr/h

5. 5. Air volumetric flow rate: 5ltr/min (0.3m3/h) Vacuum in K2: -0,2 bar rel. set the CO2 gas volumetric flow rate. CO2 gas volumetric flow rate: 1,2ltr/min at a pressure of +0.8 bar abs. Table 1.1: Concentration values of CO_2:

	Series of measurements (vol.%)							Mean values
	1	2	3	4	5	6	7	φ_E (%)
(	5 min	10 min	15 min	20 min	25 min	30 min		------------
S1								
S2								
S3								

REFERENCES

Geankoplis,C.J., "Separation Processes and Unit Operations, 4th edition, Prentice-Hall Inc., New Jersey, 2004

Appendix:

Notations/ Table of symbols in Figure 1:

A1	Cooling unit
A2	Heater before desorber inlet
K1	Absorption column (Absorber)
K2	Regeneration column (Desorber)
P1	Circulation pump for delivery to the absorption column
P2	Circulation pump for delivery to the regeneration column
P3	Circulation pump in cooling circuit
P4	Vacuum water jet pump
V	Compressor for air
W2/W3	Heat exchanger
W4	Heat exchanger (recuperator)
V1	CO_2 control valve
V2	Air control valve
V3	Non-return valve
V4	Ball valve for gas sampling at base of column
V5	Ball valve for gas sampling at centre of column
V6	Ball valve for gas sampling at head of column
V7	Ball valve
V8	Ball valve for releasing the pressure measuring point in the centre of the column
V9	Ball valve for releasing the pressure measuring point at the head of the column
V10	Control valve for adjusting the column pressure
V11	Safety valve on absorption column
V12	Control valve for adjusting the water flow rate
V13	Ball valve for liquid sampling from absorption column
V14	Ball valve for draining the water cooling circuit
V15	Ball valve for sampling regenerated absorbent
V16	Solenoid control valve for regulating the level in the absorption column K1
V17	Control valve for absorbent before regeneration column K2
V18	Non-return valve
V19	Ball valve for equalising the vacuum in column K2 after the experiment
V20	Control valve for adjusting the water quantity for the vacuum water jet pump
V21	Control valve for adjusting the quantity of stripping air
V22	Solenoid valve for filling the columns (K1&K2) with water
V23	Ball valve for manually blocking the water supply connection
V24	Ball valve for draining the regeneration column
V25	Ball valve for releasing the pressure measuring point at the base of the column

EXPERIMENT-10

Distillation of binary mixtures (Ethanol and water) by Simple/batch distillation

Aim of the experiment

Determine the composition of the ethanol in vapor and liquid phase by measuring the refractive index of the mixture.

OBJECTIVE:

To check the concentrations (compositions) of liquid-vapour phases from specific gravities and plot T-x-y diagram

Introduction and Theoretical background

The function of distillation is to separate, by vaporization, a liquid mixture of miscible and volatile substances into individual components or, in some cases, into groups of components. The separation of a mixture of alcohol and water into its components; of liquid air into nitrogen, oxygen, argon; and of crude petroleum into gasoline, kerosene, fuel oil, and lubricating stock are examples of distillation. Suppose there are two components, A and B. Both of these components are found in both phases. There are four variables: pressure, temperature, and concentrations of component A in the liquid and vapor phases (the concentrations of component B are unity less those concentrations of A). If the pressure is fixed, only one variable, e.g., liquid-phase concentration, can be changed independently and temperature and vapor-phase concentration follow. In practice, distillation may be carried out either of two principal methods. The first method is based on the production of a vapor by boiling the liquid mixture to be separated and condensing the vapors without allowing any liquid to return to the still. There is then no reflux, which is called batch distillation. [1-2] The second method is based on the return of part of the condensate to the still under such conditions that this returning liquid is brought into intimate contact with the vapors on their way to the condenser. Either of these methods may be conducted as a continuous steady-state distillation process, including single-stage partial vaporization without reflux (flash distillation) and continuous distillation with reflux (rectification), for systems containing only two components. The diagram of our system is given in **Fig 1.** [3-4]

In this system it can be investigated the principles that rule the mass and energy transference, as well as determine the optimal operation point to carry out a big quantity of separations. Distillation system is basically composed by a boiler on which two types of interchangeable columns can be adapted (plate columns and Raschig Rings column), a reflux system and a tank for the distillation. · The steam that goes to the head of the column is sent to a total condenser. · The cooling water flow that crosses the condenser is regulated and indicated by the flow meter. · The pressure loss in the column can be measured with a manometer. · The temperatures of the system are measured by temperature sensors placed in strategic positions. [5-6]

Description of experimental apparatus
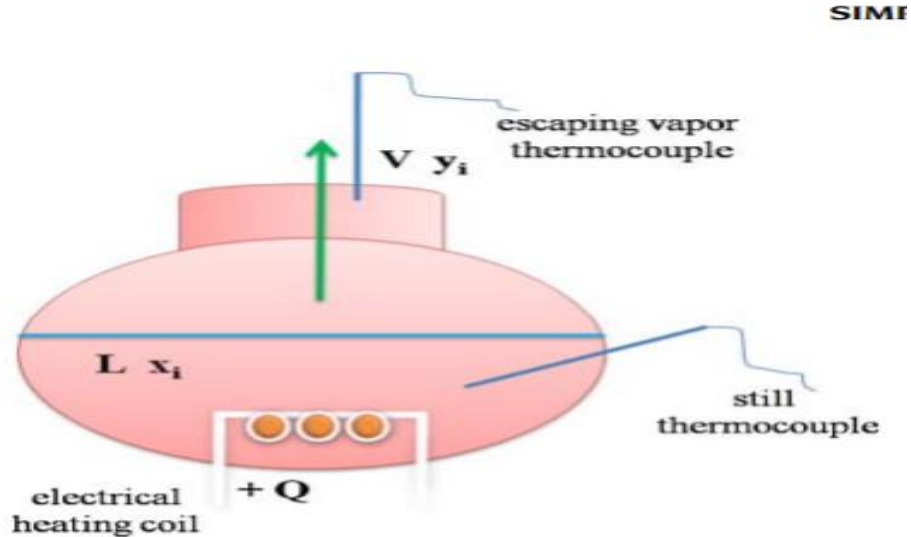
Fig 1: Simple distillation set up

Experimental Procedure

- Fill the tank with 200 ml of Ethanol and 200 ml of Water composition (50% each)
- Start the cooling water flow rate into the condenser and open the maximum flow
- Turn on the heater and let the mixture boil at boiling points range
- Change the composition of ethanol -water mixture by varying from different composition starting from 50% each to pure ethanol to get the T-x-y plot.
- Analyze the concentrations of distillate (y) from the top collection tank and condensate (x) from bottom of the feed tank.

Tabular column & worksheets

Ethanol and water composition	Boiling point temperature range		T, Specific gravity of the of the distillate (Top Product)		T, Specific gravity of the of the Residue/Condensate (Bottom product)		Composition of Distillate [TOP PRODUCT] (y) Ethanol		Composition of Condensate [BOTTOM PRODUCT] (x) water	
-------------	Start	End	T	ρ/ρ_w	T	ρ/ρ_w	Mass fraction	Mole fraction	Mass fraction	Mole fraction
50-50% each										
10-90% (Eth +water)										
20-80% (Eth +water)										
30-70%(Eth +water)										
40-60% (Eth +water)										
100% ethanol (pure)										

WORK SHEETS

Boiling point of the feed =

Temperature of distillate and Residue =

Distillate collected for every sample: 100 ml

Plots

Plot T-X-Y diagram after getting the readings of T, x, y
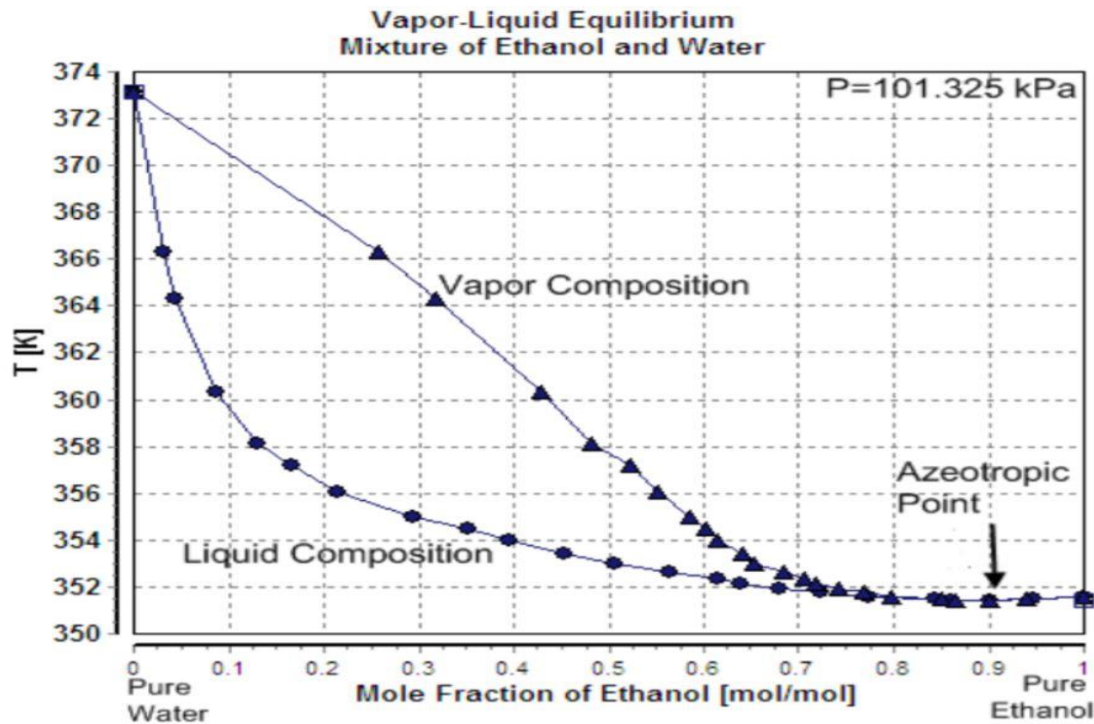
Fig 2 T-x-y diagram of ethanol water mixtures at 1 atm pressure

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1. J. H. Perry, Ed., Chemical Engineer's Handbook, 7th ed., McGraw-Hill Publishing Co., New York, NY,1999.

2. W. L. McCabe and J. C. Smith, Unit Operations of Chemical Engineering, 6th ed., McGraw-Hill Publishing Co., New York, NY., 2001.

3. C.,J.,Geankoplis,Transport Processes and Unıt Operation, 3th ed., Prantice-Hill İnternational, Canada, 1993.

4. Edibon Technical Teaching Equipment, URLhttp://www.edibon.com/products/?area=chemicalengineering&subarea=chemicaleng ineeringba sic(Visit , 07.10.2016

5. CE Gunt Experiment Manual**.**

6. Rachael L. Baumann"Solving Mass Balances on a Distillation Column" http://demonstrations.wolfram.com/SolvingMassBalancesOnADistillationColumn/ Wolfram Demonstrations Project Published: June 22 2018

TABLE I $\varrho = \varrho(p, t)$

p\t	20	21	22	23	24	25	26	27	28	29	30
0	998,20	997,99	997,77	997,54	997,29	997,04	996,78	996,51	996,23	995,94	995,65
1	996,31	996,10	995,88	995,64	995,40	995,15	994,89	994,62	994,34	994,05	993,75
2	994,49	994,27	994,05	993,82	993,57	993,32	993,06	992,78	992,50	992,21	991,90
3	992,73	992,51	992,29	992,05	991,80	991,55	991,28	991,00	990,72	990,42	990,12
4	991,02	990,81	990,58	990,34	990,09	989,83	989,56	989,28	988,99	988,69	988,38
5	989,38	989,15	988,92	988,68	988,42	988,15	987,88	987,59	987,30	986,99	986,68
6	987,78	987,55	987,31	987,06	986,80	986,53	986,25	985,95	985,65	985,34	985,02
7	986,24	986,00	985,75	985,49	985,22	984,94	984,65	984,36	984,05	983,73	983,40
8	984,73	984,49	984,23	983,96	983,68	983,40	983,10	982,79	982,48	982,15	981,82
9	983,27	983,01	982,75	982,47	982,18	981,89	981,58	981,26	980,94	980,60	980,26
10	981,85	981,58	981,30	981,01	980,71	980,41	980,09	979,76	979,43	979,08	978,73
11	980,46	980,18	979,89	979,58	979,27	978,95	978,63	978,29	977,94	977,59	977,22
12	979,10	978,80	978,50	978,18	977,86	977,53	977,18	976,83	976,47	976,11	975,73
13	977,76	977,45	977,13	976,80	976,46	976,12	975,76	975,40	975,02	974,64	974,25
14	976,44	976,11	975,78	975,43	975,08	974,72	974,35	973,97	973,58	973,19	972,78
15	975,13	974,79	974,44	974,08	973,71	973,33	972,94	972,55	972,15	971,74	971,32
16	973,83	973,47	973,11	972,73	972,34	971,95	971,54	971,13	970,71	970,29	969,85
17	972,54	972,16	971,77	971,38	970,97	970,56	970,14	969,71	969,28	968,83	968,38
18	971,24	970,84	970,44	970,02	969,60	969,17	968,73	968,29	967,83	967,37	966,90
19	969,93	969,51	969,09	968,66	968,22	967,77	967,31	966,85	966,38	965,90	965,41
20	968,61	968,17	967,73	967,28	966,82	966,35	965,88	965,40	964,91	964,41	963,91
21	967,27	966,81	966,35	965,88	965,40	964,91	964,42	963,92	963,42	962,90	962,38
22	965,90	965,43	964,94	964,45	963,96	963,45	962,94	962,43	961,90	961,37	960,83
23	964,51	964,02	963,51	963,01	962,49	961,97	961,44	960,90	960,36	959,81	959,26
24	963,09	962,58	962,05	961,53	960,99	960,45	959,91	959,35	958,79	958,23	957,66
25	961,63	961,10	960,56	960,02	959,46	958,91	958,34	957,77	957,19	956,61	956,02
26	960,14	959,59	959,03	958,47	957,90	957,32	956,74	956,16	955,56	954,96	954,36
27	958,61	958,04	957,47	956,89	956,30	955,71	955,11	954,51	953,90	953,28	952,66
28	957,05	956,46	955,86	955,27	954,66	954,05	953,44	952,82	952,22	951,57	950,93
29	955,44	954,83	954,22	953,61	952,99	952,36	951,73	951,10	950,46	949,82	949,17
30	953,78	953,16	952,54	951,91	951,27	950,63	949,99	949,34	948,69	948,03	947,37
31	952,09	951,46	950,82	950,17	949,52	948,87	948,21	947,55	946,88	946,21	945,53
32	950,36	949,71	949,05	948,39	947,73	947,06	946,39	945,72	945,04	944,35	943,67
33	948,58	947,92	947,25	946,58	945,90	945,22	944,54	943,85	943,16	942,47	941,77
34	946,77	946,09	945,41	944,73	944,04	943,35	942,65	941,95	941,25	940,54	939,83
35	944,93	944,23	943,54	942,84	942,14	941,44	940,73	940,02	939,31	938,59	937,87
36	943,03	942,33	941,63	940,92	940,21	939,49	938,78	938,06	937,33	936,61	935,88
37	941,11	940,40	939,68	938,96	938,24	937,52	936,79	936,06	935,33	934,59	933,86
38	939,15	938,43	937,71	936,98	936,25	935,51	934,78	934,04	933,30	932,55	931,81
39	937,16	936,43	935,70	934,96	934,22	933,48	932,74	931,99	931,24	930,49	929,73
40	935,15	934,41	933,66	932,92	932,17	931,42	930,67	929,92	929,16	928,40	927,64
41	933,10	932,35	931,60	930,85	930,10	929,34	928,58	927,82	927,06	926,29	925,52
42	931,03	930,28	929,52	928,76	928,00	927,24	926,47	925,70	924,93	924,16	923,38
43	928,94	928,18	927,41	926,65	925,88	925,11	924,34	923,56	922,79	922,01	921,22
44	926,82	926,06	925,29	924,52	923,74	922,96	922,19	921,41	920,62	919,84	919,05
45	924,69	923,92	923,14	922,36	921,58	920,80	920,02	919,23	918,44	917,65	916,86
46	922,53	921,76	920,98	920,19	919,41	918,62	917,83	917,04	916,25	915,45	914,65
47	920,37	919,58	918,80	918,01	917,22	916,43	915,64	914,84	914,04	913,24	912,44
48	918,18	917,39	916,60	915,81	915,02	914,22	913,42	912,62	911,82	911,01	910,21
49	915,98	915,19	914,40	913,60	912,80	912,00	911,20	910,39	909,59	908,78	907,96
50	913,77	912,98	912,18	911,38	910,57	909,77	908,96	908,15	907,34	906,53	905,71

Tab. 6.1   For the ethanol/water mixture: Density ρ in kg/m³ as a function of the ethanol mass fraction p and temperature t , 0 ≤ p ≤ 50% , 20 ≤ t ≤ 30°C

TABLE I　　$\varrho = \varrho(p, t)$

p / t	20	21	22	23	24	25	26	27	28	29	30
50	913,77	912,98	912,18	911,38	910,57	909,77	908,96	908,15	907,34	906,53	905,71
51	911,55	910,75	909,95	909,14	908,34	907,53	906,72	905,90	905,09	904,27	903,45
52	909,31	908,51	907,70	906,90	906,09	905,27	904,46	903,64	902,82	902,00	901,18
53	907,07	906,26	905,45	904,64	903,83	903,01	902,19	901,37	900,55	899,73	898,90
54	904,81	904,00	903,19	902,38	901,56	900,74	899,92	899,10	898,27	897,44	896,61
55	902,55	901,74	900,92	900,10	899,28	898,46	897,64	896,81	895,98	895,15	894,31
56	900,28	899,46	898,64	897,82	897,00	896,17	895,35	894,52	893,68	892,85	892,01
57	897,99	897,18	896,35	895,53	894,70	893,88	893,05	892,21	891,38	890,54	889,70
58	895,70	894,88	894,06	893,23	892,40	891,57	890,74	889,90	889,06	888,22	887,38
59	893,40	892,58	891,75	890,93	890,09	889,26	888,42	887,59	886,74	885,90	885,06
60	891,10	890,27	889,44	888,61	887,78	886,94	886,10	885,26	884,42	883,57	882,72
61	888,78	887,95	887,12	886,29	885,45	884,61	883,77	882,93	882,08	881,23	880,38
62	886,46	885,63	884,79	883,96	883,12	882,28	881,43	880,59	879,74	878,89	878,04
63	884,13	883,30	882,46	881,62	880,78	879,94	879,09	878,24	877,39	876,54	875,68
64	881,79	880,96	880,12	879,28	878,43	877,59	876,74	875,89	875,04	874,18	873,32
65	879,45	878,61	877,77	876,92	876,08	875,23	874,38	873,53	872,67	871,82	870,96
66	877,09	876,25	875,41	874,56	873,72	872,87	872,01	871,16	870,30	869,44	868,58
67	874,73	873,89	873,04	872,20	871,35	870,50	869,64	868,78	867,93	867,07	866,20
68	872,37	871,52	870,67	869,82	868,97	868,12	867,26	866,40	865,54	864,68	863,82
69	869,99	869,14	868,29	867,44	866,59	865,73	864,88	864,02	863,15	862,29	861,42
70	867,61	866,76	865,91	865,06	864,20	863,34	862,48	861,62	860,76	859,89	859,02
71	865,22	864,37	863,52	862,66	861,80	860,94	860,08	859,22	858,35	857,49	856,62
72	862,83	861,97	861,12	860,26	859,40	858,54	857,68	856,81	855,94	855,07	854,20
73	860,43	859,57	858,71	857,85	856,99	856,13	855,26	854,40	853,53	852,66	851,78
74	858,02	857,16	856,30	855,44	854,58	853,71	852,84	851,98	851,11	850,23	849,36
75	855,60	854,74	853,88	853,02	852,15	851,29	850,42	849,55	848,68	847,80	846,93
76	853,17	852,31	851,45	850,59	849,72	848,85	847,98	847,11	846,24	845,36	844,49
77	850,74	849,88	849,02	848,15	847,28	846,41	845,54	844,67	843,79	842,92	842,04
78	848,30	847,44	846,57	845,71	844,84	843,97	843,09	842,22	841,34	840,46	839,58
79	845,85	844,98	844,12	843,25	842,38	841,51	840,63	839,76	838,88	838,00	837,12
80	843,39	842,52	841,65	840,78	839,91	839,04	838,17	837,29	836,41	835,53	834,65
81	840,91	840,05	839,18	838,31	837,44	836,56	835,69	834,81	833,93	833,05	832,16
82	838,43	837,56	836,69	835,82	834,95	834,07	833,20	832,32	831,44	830,55	829,67
83	835,93	835,06	834,19	833,32	832,44	831,57	830,69	829,81	828,93	828,05	827,16
84	833,41	832,54	831,67	830,80	829,93	829,05	828,17	827,29	826,41	825,53	824,64
85	830,90	830,01	829,14	828,26	827,39	826,51	825,64	824,76	823,87	822,99	822,10
86	828,32	827,45	826,58	825,71	824,84	823,96	823,08	822,20	821,32	820,44	819,55
87	825,75	824,88	824,01	823,14	822,26	821,39	820,51	819,63	818,75	817,87	816,98
88	823,15	822,28	821,41	820,54	819,67	818,79	817,92	817,04	816,16	815,27	814,39
89	820,53	819,66	818,79	817,92	817,05	816,17	815,30	814,42	813,54	812,66	811,77
90	817,88	817,01	816,15	815,27	814,40	813,53	812,65	811,77	810,90	810,02	809,13
91	815,21	814,34	813,47	812,60	811,73	810,85	809,98	809,10	808,23	807,35	806,47
92	812,49	811,63	810,76	809,89	809,02	808,15	807,27	806,40	805,52	804,65	803,77
93	809,75	808,88	808,02	807,15	806,28	805,41	804,54	803,66	802,79	801,91	801,04
94	806,97	806,10	805,23	804,37	803,50	802,63	801,76	800,89	800,02	799,15	798,27
95	804,14	803,28	802,41	801,55	800,68	799,81	798,94	798,07	797,21	796,34	795,47
96	801,27	800,41	799,55	798,68	797,82	796,95	796,08	795,22	794,35	793,48	792,61
97	798,36	797,49	796,63	795,77	794,90	794,04	793,17	792,31	791,44	790,58	789,71
98	795,38	794,52	793,66	792,80	791,94	791,07	790,21	789,35	788,48	787,62	786,76
99	792,35	791,49	790,63	789,77	788,91	788,05	787,19	786,33	785,46	784,60	783,74
100	789,24	788,38	787,53	786,67	785,81	784,95	784,09	783,23	782,37	781,51	780,65

Tab. 6.2　　For the ethanol/water mixture: Density ρ in kg/m³ as a function of the ethanol mass fraction p and temperature t , 50 ≤ p ≤ 100% , 20 ≤ t ≤ 30°C

6.1.2 Determining concentration by density and temperature

The object of density measurement is to determine the concentration of the mixture.
Common concentrations quoted for the ethanol/water mixture are the ethanol volumetric fraction and mass fraction.

Any indication of the volumetric fraction is only unambiguously specific in conjunction with the associated reference temperature. So a more suitable quantity for comparison and balancing purposes is the **ethanol mass fraction in the mixture**.

To determine this mass fraction, the tables from Chapter 6.1.1 can be used.

A comparison of the densities at different temperatures shows that the concentration is substantially dependent on the sample temperature as well as the density.
If possible, the **samples** should be appropriately **temperature-controlled** prior to measuring the density, such as to the reference temperature 20 °C. Then the concentration can be read directly from the table with the measured density at the relevant temperature.

No such temperature control was available for the experiments on which this chapter is based. Consequently, the density and temperature of the sample were measured for each concentration calculation. Allowance is made for the influence of the temperature by means of linear interpolation (see Evaluation, Chapter 6.3.5.2, Page 82 ff).
The major influence of small changes in density on the ethanol mass fraction is illustrated in Chapter 6.3.6, Page 89 ff. under item 5., based on a concrete example.

Experiment-11

Liquid -Liquid Extraction

Objective of the extraction

The main objective of **liquid-liquid extraction** is to transfer **a transition component** from a liquid mixture into a liquid solvent

Designation of the substances involved

The simplest form of **liquid-liquid extraction** involves three liquids:

Transition component -------- Ethanol

Carrier liquid---------------------Vegetable oil

Solvent----------------------- -----Tap water

Phenomena of Liquid-Liquid Extraction & Theory

The liquid mixture of the transition component and carrier liquid is called the **feed**. In the feed, the **transition component** is dissolved in the **carrier liquid**. **Carrier liquid** and **solvent** together form the **sol- vent system**. The transition component must be soluble in both the carrier liquid and the solvent.

The carrier liquid and solvent should together form a **phase boundary** in order to allow the separation of the two phases. Therefore, carrier liquid and solvent should be as mutually insoluble in each other as possible. The two **phases**, which we get at the end of **liquid-liquid extraction** are called the **extract** and the **raffinate**. The **extract** is essentially a solution of the transition component in the original solvent. The **raffinate** is essentially the original feed, less the extracted proportion of the transition component.

SETTING THE PHASE BOUNDARY

A **phase boundary** forms at the point where the mutually insoluble solvents (**carrier liquid** and **solvent**) meet.

If the phase boundary moves up or down in the extraction column, then this will result in unwanted breaks. In other words, either FEED enters the solvent/the extract, or solvent enters the feed/the raffinate. The aim is to keep the phase boundary at a similar level during the entire extraction time to enable reproducible experiments.

Setting the phase boundary during the experiment requires some practice. It requires an understanding of the hydraulic system. A key component of the hydraulic system is the extraction column K1(9). The phase boundary is determined by the interaction of the two incoming and two outgoing material flows.

Simplified representation of liquid-liquid extraction

The illustration in symbolizes **liquid-liquid extraction** in a beaker, with the following simplifications:

Only three liquids are involved.

The transition component transfers completely from the carrier liquid into the solvent.

On the left is the beaker with two liquid phases. **Carrier liquid** and **solvent** together form the **sol- vent system**. The carrier liquid and solvent together form a **phase boundary**. They are insoluble in one another. This is the condition so that separation into two phases can occur after the actual extraction. Also required is a clear density difference between the carrier liquid and solvent as shown in **fig 1**
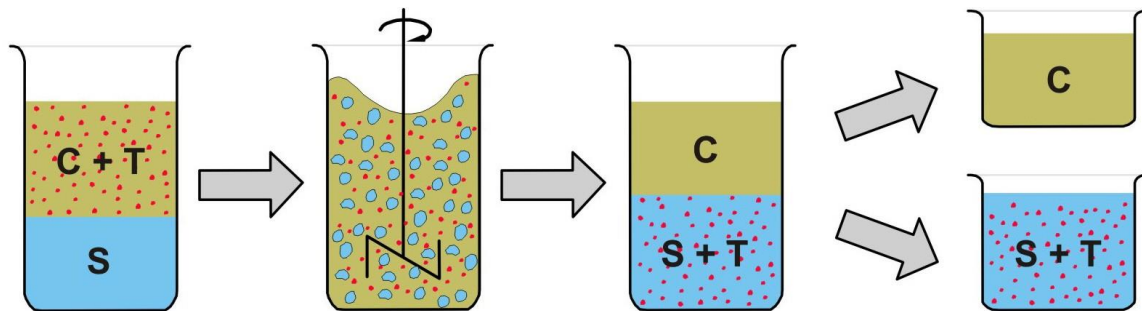
Liquids involved	Abbreviation
Transition component	T
Carrier liquid	C
Solvent	S

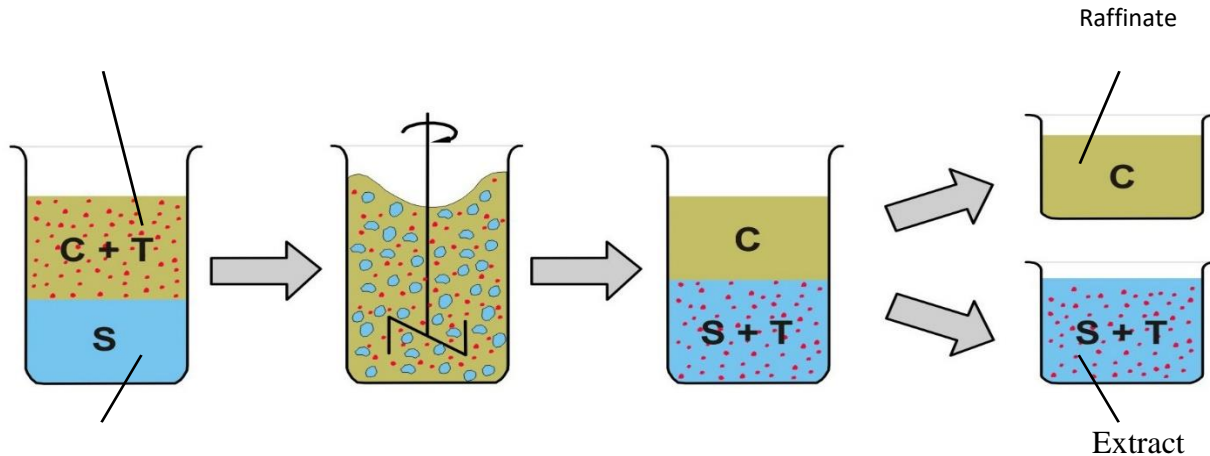**Fig 1: Beaker experiments with showing carrier liquid and solvent**

Fig 2: Liquid-liquid extraction, identification of incoming and outgoing materials

The following terms are common in **liquid-liquid extraction**:

The liquid mixture of the transition component and carrier liquid is called the **feed**.

The two liquid phases, which we get at the end of **liquid-liquid extraction** are called the **extract** and the **raffinate**.

The **extract** is essentially a solution of the transi-tion component in the original solvent. The **raffinate** is essentially the original feed, less the extracted proportion of the transition component.

This results in:

From solvent and feed we get raffinate and extract, as shown in **Fig 2**

The aim of liquid-liquid extraction is to trans- form as high a proportion of the transition component from the feed into the extract as possible.

Process Flowsheet

The **fig 3** shows the process diagram for the **CE 620 liquid-liquid extraction** unit. From the top of the extraction column the feed returns to the feed tank (**feed return**).The solvent is continuously pumped from the **solvent tank** B1(13) by the **solvent pump** P1(17) into the top of the extraction column (**solvent inflow**). The solvent moves downwards in **counterflow** to the feed. From the bottom of the extrac- tion column, the solvent returns to the solvent tank (**solvent return**).

The driving force for the counterflow is the **density difference** between solvent and feed. Therefore, the material and/or the mixture with the lower density must be pumped into the bottom of the extraction column. **Fig 4** shows the front view of LLE set up.
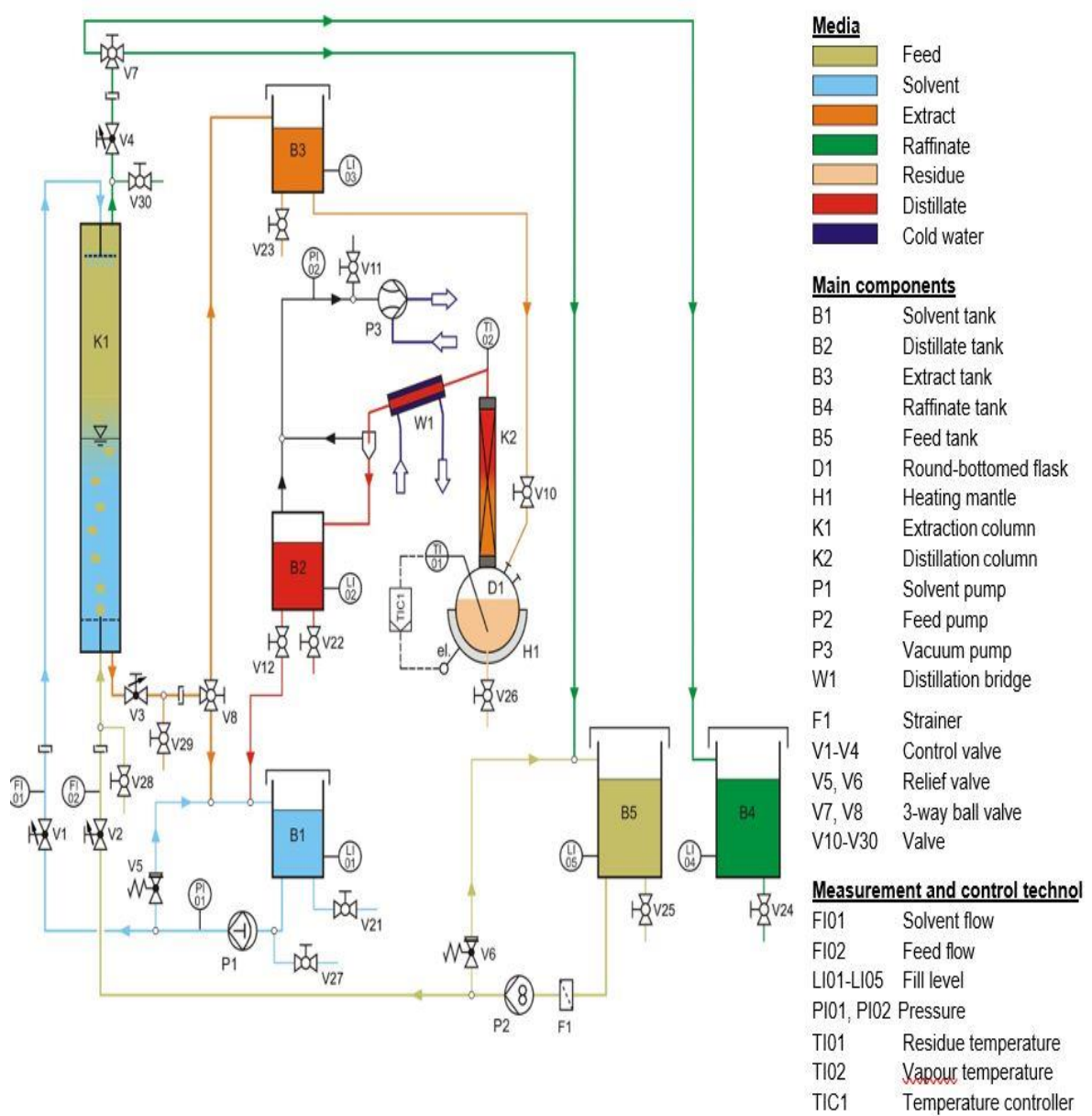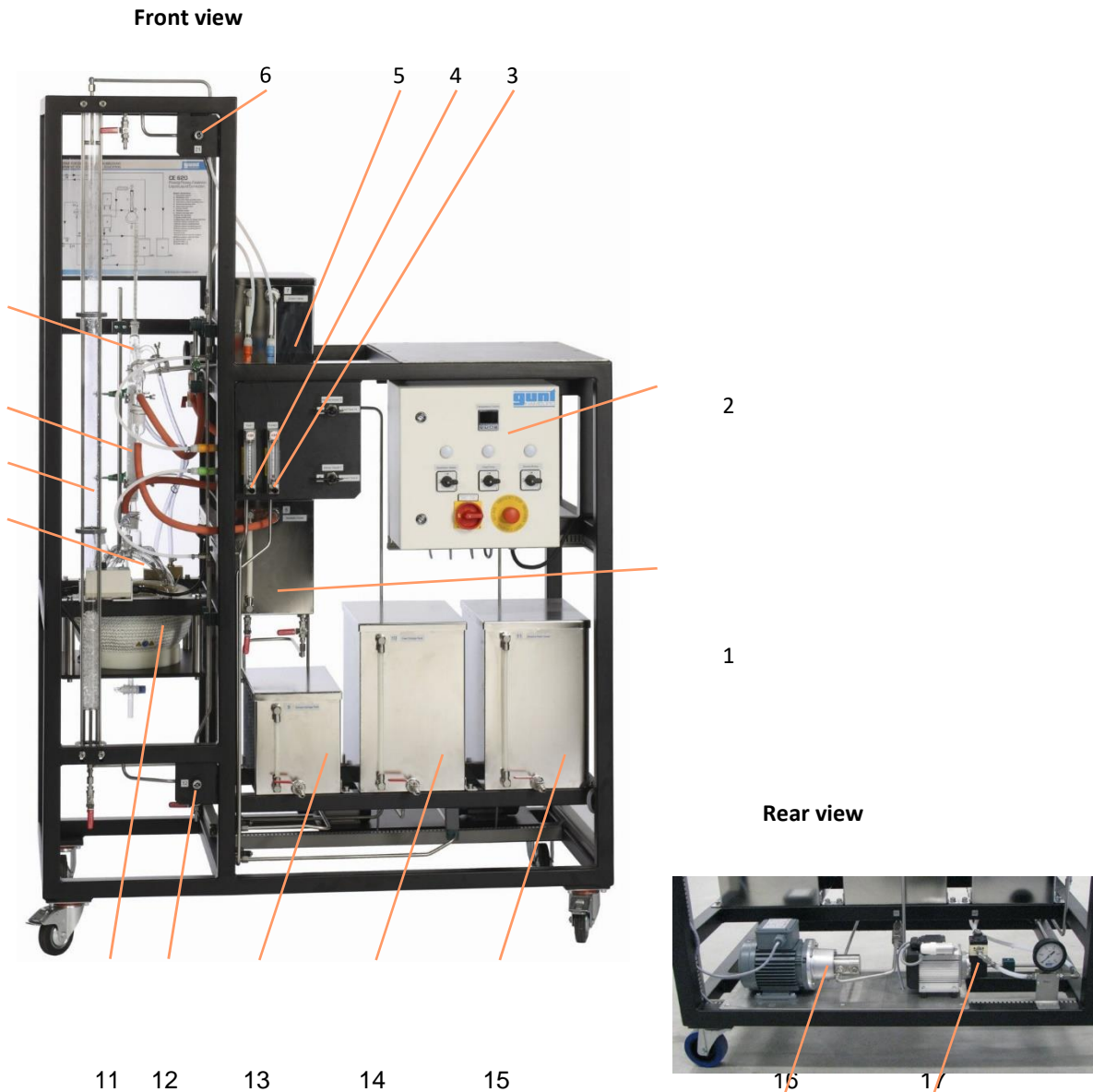

Media

	Feed
	Solvent
	Extract
	Raffinate
	Residue
	Distillate
	Cold water

Main components

B1	Solvent tank
B2	Distillate tank
B3	Extract tank
B4	Raffinate tank
B5	Feed tank
D1	Round-bottomed flask
H1	Heating mantle
K1	Extraction column
K2	Distillation column
P1	Solvent pump
P2	Feed pump
P3	Vacuum pump
W1	Distillation bridge
F1	Strainer
V1-V4	Control valve
V5, V6	Relief valve
V7, V8	3-way ball valve
V10-V30	Valve

Measurement and control technol

FI01	Solvent flow
FI02	Feed flow
LI01-LI05	Fill level
PI01, PI02	Pressure
TI01	Residue temperature
TI02	Vapour temperature
TIC1	Temperature controller

Fig 3: Process flow sheet of extraction Column

Device design

Front view

Rear view

1	Distillate tank (B2)	11	Heating mantle (H1)
2	Control cabinet	12	Regulating valve for solvent return (V3)
3	Regulating valve for solvent inflow (V1)	13	Solvent tank (B1)
4	Regulating valve for feed inflow (V2)	14	Feed tank (B5)
5	Extract tank (B3)	15	Raffinate tank (B4)
6	Regulating valve for feed return (V4)	16	Feed pump (P2)
7	Distillation bridge (W1)	17	Solvent pump (P1)
8	Distillation column (K2)		
9	Extraction column (K1)		
10	Round-bottomed flask (D1)		

Fig. 4   Overview of the CE 620 liquid-liquid extraction system (Front view)

LLE for batch mode- without distillation

The mass transfer of liquid-liquid extraction takes place in the **extraction column** K1(9). This is where **feed** and **solvent** meet. When this hap- pens, a part of the **transition component** is extracted from the feed into the solvent.

The feed is continuously pumped from the **feed tank** B5(14) by the **feed pump** P2(16) into the bottom of the extraction column (**feed inflow**). The feed moves upwards in the extraction column. **Fig 5** shows the Counter current extraction column
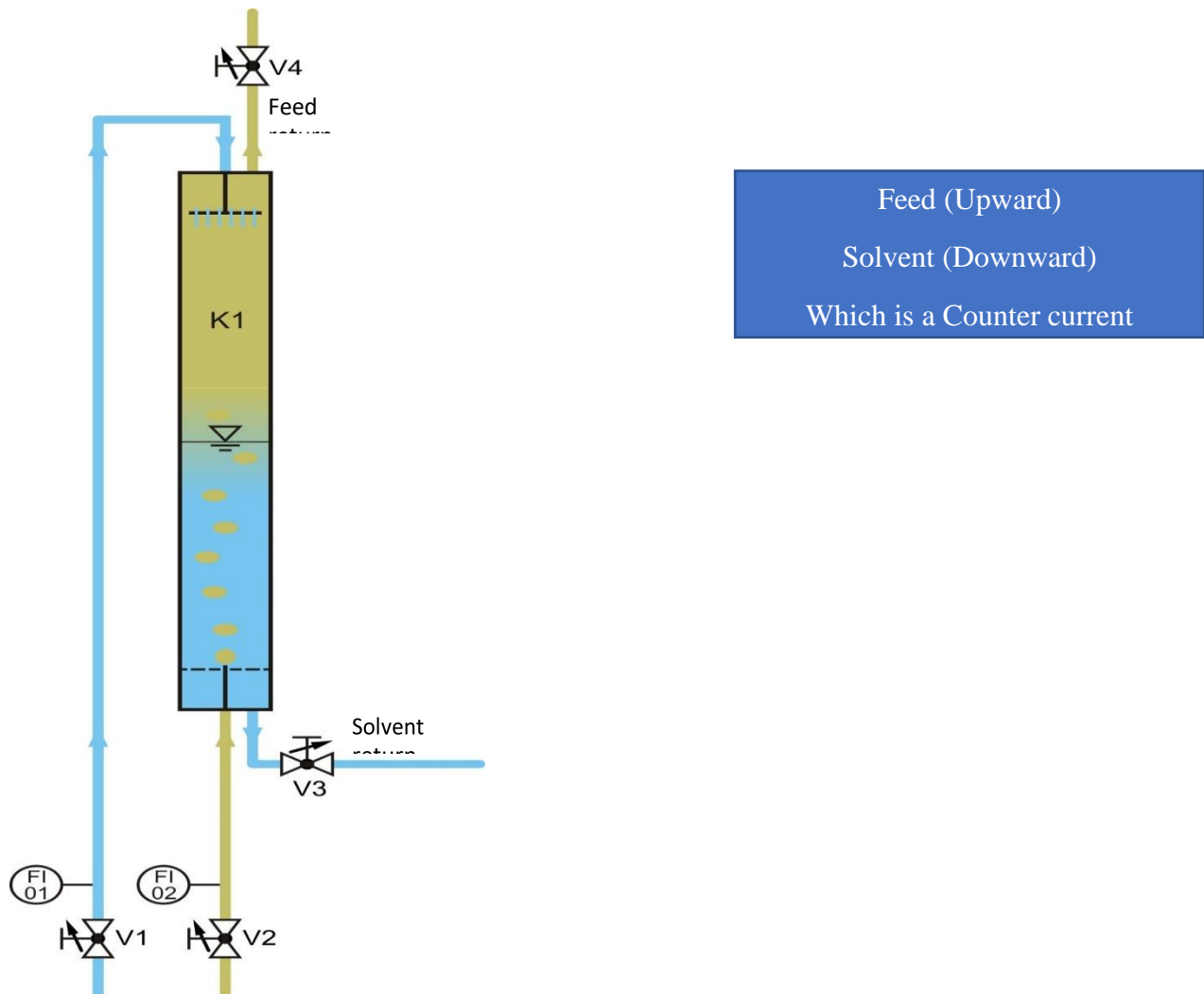

Fig 5: Counter current extraction column

When unthrottled and with free run-off, the solvent pump delivers with maximum flow rate. The flow rate is reduced by the counter pressure in the extraction column K1. In addition, flow can

be throttled using the **regulating valve** V_1. The **relief valve** V_5 starts when the corresponding response pressure is reached. From the relief valve, the excess solvent returns to the solvent tank B1(13) via the **recirculation line**. The actual solvent flow rate is thus divided into the desired flow to the extraction column and the excess back into the solvent tank.

The flow measurement (FI01) and throttling (**regulating valve V1**) functions are combined in a single component (see page 25). FI01 is designed as a **variable-area flowmeter**. The **valve** V27 is used to vent the suction line in the event of any suction problems. The **valve** V29 is used for emptying, cleaning and rinsing the extraction column and solvent system

LLE for Continuous Mode

The below **figure 6** shows the process flow diagram for continuous mode, without distillation
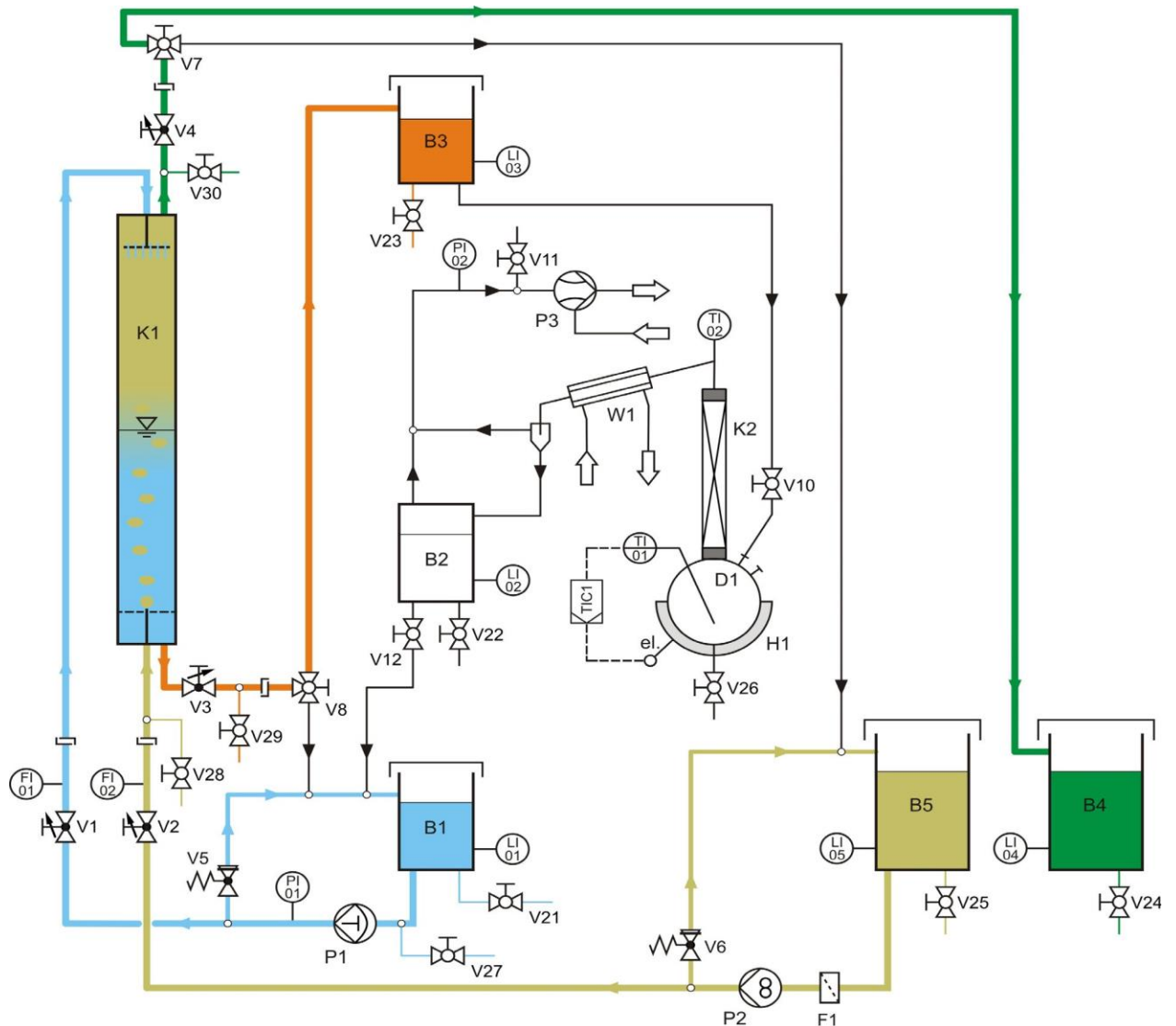
Fig 6 CE 620 Process diagram, continuous mode variant, without distillation

(It shows different feed -product separation with colour coding)

The process description is largely similar to the process description for batch mode The key difference is that in continuous mode, only a single cycle of feed and solvent through the extraction column K1(9) takes place. The solvent return goes directly into the **extract tank** B3(5) as extract. The feed return goes directly into the **raffinate tank** B4(15) as raffinate. By comparison, in batch mode multiple cycles of feed and solvent are possible. Therefore, in batch mode we usually obtain a better **mass transfer** and a higher concentration of the transition component in the extract.

EXPERIMENTS

This chapter describes experiments with the recommended **model material system** of **" extraction of ethanol from refined rapeseed oil with water."**

The experiments use:

Refined rapeseed oil as carrier liquid,

Ethanol as transition component,

Water as solvent, as well as methylated spirits, detergent and dilute acetic acid as cleaning agents.

Table 1 Materials & Cleaning agents used for extraction

Materials used	Note
Ethanol	Purity > 99,5%.
Rapeseed oil	Commercially available. Refined rapeseed oil.
Washing-up liquid	Commercially available. Notes such as "high fat dissolving power" are desired.
Methylated spirits	Commercially available. Ethanol volume fraction of about 95%.
Dilute acetic acid	Commercially available. Vinegar essence, acetic acid about 25%.

Experiments with Device- Batch Procedure

For the material model system, the liquid -liquid system has priority. At the starting of the experiment, by mixing the ethanol and rapeseed oil is used to prepare the feed

The following section describes the **standard experiment** for **liquid-liquid extraction** with **CE 620**.

The **experimental conditions** are:

Extraction of ethanol from rapeseed oil with water.

Mass fraction of ethanol in the feed 10%.

Equal masses of feed and solvent, each 5000g.

Extraction time 180min.

Solvent flow 400 mL/min.

Feed flow 800 mL/min (display value).

Conducting the experiment and cleaning

During the experiments, rapeseed oil flows through most of the components of the trainer and/or the components come into contact with the constituents of the rapeseed oil.

After contact with water and ambient air, some of the constituents of the rapeseed oil tend to undergo chemical changes. **Rapeseed oil residues** can become **rancid** and cause **disturbing odors**. In addition, if cleaning is inadequate it can lead to deposits of solids building up in the trainer, causing **malfunctions**.

It is therefore important to remove as much rapeseed oil from the trainer as possible after each experiment. The rapeseed oil residues remaining in the trainer must be removed by normal cleaning after the end of the experiment

Conducting the LIQUID-LIQUID EXTRACTION In batch mode

The solvent water required (Tap water): 5 lit

Vegetable oil: 500 ml

Ethanol: 500 ml

Remove cover from tanks B1 and B3-B5.

Connect the trainer to the mains power supply

Turn main switch to "1"

Pour 5000 ml of tap water into solvent tank B1

Pour 500g of ethanol into feed tank B5.

Quickly add 500 ml of Vegetable oil into the feed tank. (First add ethanol to the feed tank and then rape seed oil, otherwise the lighter ethanol would float on the top)

Start the stopwatch and stir occasionally circulate the feed for 25 minutes, then stop the stopwatch and feed pump P2.

Measure and note down the ambient temperatures of the air, Solvent and feed.

Position the 3 way ball valve so as to get the flow for batch operation (as shown in fig 3.4)

Ensure that the Valve V30 is opened.

Fully open the regulating valve V4 for feed return

Close regulating valve V3 for solvent return.

Start solvent pump P1

If there are suction problems, vent suction hose. To do this, open valve V27 briefly.

Using regulating valve V1, adjust solvent flow to about 80 mL/ min.

Start feed pump P2.

Using regulating valve V2, set feed flow to about 800 mL/min (display value, not actual flow).

Therefore, the phase boundary will form at the start of extraction at approximately half the height of the extraction column.

- Check that the feed flow is about 800 mL/min. Readjust with V2 if it is different.
- Measure the position of the phase boundary, based on half the extraction height (see Fig. 3.16, Page 33), using a ruler or tape measure.
- Note down position of the phase boundary and opening of V3 on the worksheet.
- At regular intervals, for example after every 10min, re-record the values and note down on the worksheet, including information on the time.
- By comparing with the previous value on the worksheet, determine whether the phase boundary has fallen or risen.

  If the phase boundary deviates significantly from half the extraction height, adapt the opening of regulating valve V3 for solvent return:

  – **If the phase boundary is falling, close regulating valve V3 a little more.**

  – **If the phase boundary is rising, open regulating valve V3 a little more.**

  Note down new position of the regulating valve V3 on the worksheet

Repeat density measurement of the solvent after 20min, and for the last time after 180min of extraction time.

After 180min of extraction time, stop pumps P1 and P2.

This is the **extraction end**.

Close regulating valves V1- V4.

Open valve V30.

Now the oil phase settles on the water phase.

By opening the regulating valve V3, the solvent from the extraction column can flow back into solvent tank B1.

Close V3 as soon as the phase boundary reaches the base of the extraction column.

By opening valve V28, pour feed from the extraction column into the measuring beaker (2L).

Then, by opening valve V29, the remaining feed can flow from the extraction column into the measuring beaker.

Drain measuring beaker into the feed tank B5.

Now, **extract** is in the solvent tank and **raffinate** is in the feed tank (see Chapter 3.4.1.1, Page 20ff).

In order to make it easier to study the mass balance, determine the **total mass of raffinate**. To do so:

Let the raffinate flow from feed tank B5 into a tare weighted measuring beaker.

Determine the net mass of raffinate and note down on the worksheet.

Empty measuring beaker into a separate vessel, for example a metal can.

Repeat until the feed tank B5 is empty.

Finally, drain the raffinate from the feed inflow line into the tare-weighted measuring beaker. To do this, disconnect the feed inflow from the quick coupling. Then attach hose to quick coupling from the accessories, and affix the quick coupling (see photo opposite).

Hold hose in the measuring beaker and start feed pump P2.

Stop pump P2 after emptying the feed inflow line.

Determine the net mass of raffinate and note down on the worksheet.

In order to make it easier to study the mass bal- ance, determine the **total mass of extract**. To do so:

Let the extract flow from solvent tank B1 into a tare-weighted measuring beaker.

Determine the net mass of extract and note down on the worksheet.

Repeat until the solvent tank B1 is empty.

Finally, drain the extract from the solvent inflow line into the tare-weighted measuring beaker. To do this, start solvent pump P1.

Stop P1 as soon as solvent stops entering the extraction column.

By opening valve V29, the remaining extract can flow into the tare-weighted measuring beaker. Determine the net mass of extract and note down on the worksheet.

This concludes the actual experiment.

Never leave the trainer in an uncleaned state.

Be sure to perform regular cleaning after the end of the experiment,

Cleaning Procedures: (Refer page 100 to 105 for cleaning the equipment and finally turn Main Switch to Zero.

MEASUREMENTS

At the start of extraction 10kg of liquid were added to the trainer (sum of solvent and feed).

At the end of extraction approximately 9,2kg of liquid were removed from the trainer (sum of extract and raffinate).

A large part of the measured values were entered by hand into a copy of the worksheet Tab. 6.3, Page 119 during the experiment (measured values for the phase boundary and for the density measurement).

$\Box t$ Extr Extrac-tion time min	$\dot{Feed}$ V mL / min	$\dot{V}_{Solv}$ mL /min	V4 Opening %	Phase boundary V3 Opening, chg to... Turns	Level Phase boundary cm	Notes
0	800	400	100	1,5	+2	Start of extraction
3	800	400	100	1,0	-5	
6	800	400	100	1,1	+1	
8	800	400	100	1,2	+4	
16	800	400	100	1,3	+3	
26	800	400	100	1,2	-4	
39	800	400	100	1,1	-16	
51	800	400	100	1,0	-23	
59	800	400	100	(1,0)	-14	
66	800	400	100	(1,0)	-10	
78	800	400	100	(1,0)	-3	
86	800	400	100	1,1	+11	
93	800	400	100	1,2	+12	

105	800	400	100	(1,2)	+10	
116	800	400	100	(1,2)	+7	Phase boundary expansion
136	800	400	100	(1,2)	-3	
146	800	400	100	1,1	-9	
161	800	400	100	1,0	-13	
173	800	400	100	(1,0)	-5	
180	800	400	100	(1,0)	+1	Extraction end

Measurement			Evaluation			Notes
$\Box t_{Extr}$ min	T °C	m /100mL g	$w_E\,(T_x)$ %	$w_E\,(T_{x+1})$ %	$w_E\,(T)$ %	
20	18,7	99,51				
40	18,7	99,39				
60	18,8	99,42				
80	18,8	99,37				
100	18,7	99,20				
120	18,5	99,20				
140	18,5	99,20				
160	18,5	99,10				
180	18,6	99,11				

Measurement			Evaluation			Notes
$\Box t_{Extr}$ min	T °C	m /100mL g	$w_E\,(T_x)$ %	$w_E\,(T_{x+1})$ %	$w_E\,(T)$ %	
20	18,7	99,51	1,88	1,78	1,8	
40	18,7	99,39	2,56	2,44	2,5	
60	18,8	99,42	2,39	2,28	2,3	
80	18,8	99,37	2,67	2,59	2,6	
100	18,7	99,20	3,66	3,55	3,6	
120	18,5	99,20	3,66	3,55	3,6	
140	18,5	99,20	3,66	3,55	3,6	
160	18,5	99,10	4,20	4,08	4,1	
180	18,6	99,11	4,20	4,08	4,1	

Worksheet

The following worksheet can be used to note down measurements you take yourself in an experiment.

The worksheet is also used to facilitate the evaluation after the experiment has ended. Fields for the mass fractions of ethanol are provided along- side the measured values for temperature and density.

$\Box t$ Extr Extraction time min	Feed $\dot{V}$ mL / min	$\dot{V}_{Solv}$ mL /min	V4 Opening %	Phase boundary V3 Opening, chg to... Turns	Level Phase boundary cm	Notes
0						Start of extraction

Measurement			Evaluation			Notes
$\Box t_{Extr}$ min	T °C	m /100mL g	$w_E\,(T_x)$ %	$w_E\,(T_{x+1})$ %	$w_E\,(T)$ %	

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