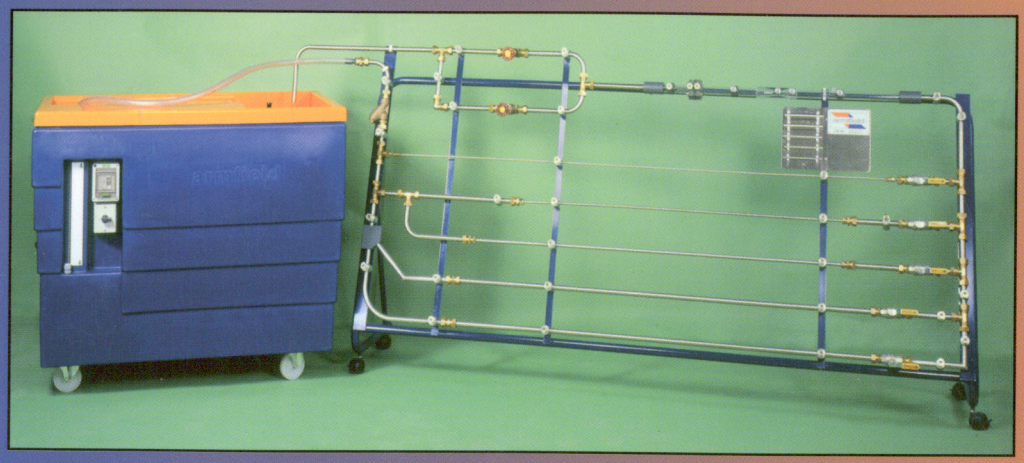

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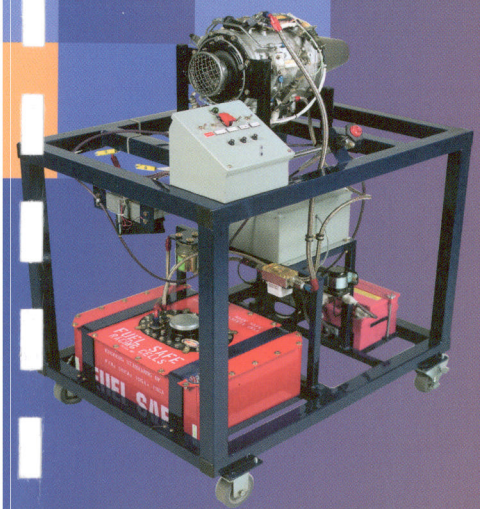
Instruction Manual



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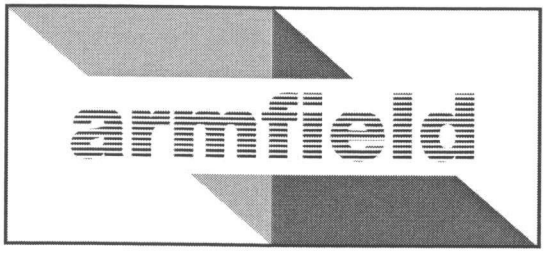

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INSTRUCTION MANUAL

UOP3CC

ISSUE 26

SEPTEMBER 2007

CONTINUOUS DISTILLATION COLUMN

IMPORTANT SAFETY INFORMATION

All practical work areas and laboratories should be covered by local safety regulations **which must be followed at all times.**

It is the responsibility of the owner to ensure that all users are made aware of relevant local regulations, and that the apparatus is operated in accordance with those regulations. If requested then Armfield can supply a typical set of standard laboratory safety rules, but these are guidelines only and should be modified as required.

Your **Continuous Distillation Column** has been designed to be safe in use when installed, operated and maintained in accordance with the instructions in this manual. As with any piece of sophisticated equipment, dangers may exist if the equipment is misused, mishandled or badly maintained.

Supervision of users should be provided whenever appropriate. Proper medical supplies relevant to the possible safety hazards must be available, with instructions for use provided.

Explosion Hazard

Operation of this equipment involves the use of highly flammable liquids or vapours.

The equipment complies with relevant British Standards for equipment being used in a Zone 1 area. This is the area within the framework of the process where "an explosive gas - air mixture is likely to occur in normal operation".

In locations governed by British regulations, the area surrounding the equipment should be designated as a Zone 2 area where no non-flameproof electrical equipment can be used. This area should extend for 2 metres around the equipment.

Other locations may have different legal and safety requirements which must be followed.

- Flammable substances must be properly stored and handled
- Keep the equipment and any flammable materials required for its use away from flames, very high temperatures and sources of sparks.
- Users must be informed of the correct procedure in the event of an emergency

Further information is available in the Specifications and Operation sections of this manual.

Electrical Safety

The equipment described in this Instruction Manual operates from a mains voltage electrical supply. It must be connected to a supply of the same frequency and voltage as marked on the equipment or the mains lead. If in doubt, consult a qualified electrician or contact Armfield.

The equipment must not be operated with any of the panels removed.

To give increased operator protection, the unit incorporates a Residual Current Device (RCD), alternatively called an Earth Leakage Circuit Breaker, as an integral part of this equipment. If through misuse or accident the equipment becomes electrically dangerous, the RCD will switch off the electrical supply and reduce the severity of any electric shock received by an operator to a level which, under normal circumstances, will not cause injury to that person.

At least once each month, check that the RCD is operating correctly by pressing the TEST button. The circuit breaker **MUST** trip when the button is pressed. Failure to trip means that the operator is not protected and the equipment must be checked and repaired by a competent electrician before it is used.

Hot Surfaces

This apparatus is capable of producing temperatures that could cause burns.

- Allow time for the equipment to cool before handling any of the components.
- Do not touch any surfaces with a 'Hot Surfaces' warning label.
- Any safety guards are there for operator protection- they must not be removed except as described in this manual, and nothing should be inserted through the guards.
- The apparatus should not be left unattended while switched on.

Hot Liquids and Gasses

This apparatus contains fluid at temperatures capable of causing scalds, and hot gas, vapour or steam at temperatures capable of causing scalds.

- Always allow time for the apparatus to cool before disconnecting any tubing.
- Avoid skin contact with hot fluids. Take particular care when performing manual collection of hot fluid during use.
- Ensure that the outlets for hot gas exiting the equipment are directed away from anything that could be harmed by high temperatures.
- Do not place anything into any gas or steam outlet or otherwise obstruct the outlet.
- Avoid skin contact with hot gas. Be aware that the heated gas stream can extend for some distance and may not be visible.

- Always operate the apparatus according to the Operation instructions described in this manual.
- Use only those fluids described in this manual when setting up and operating this equipment.

Heavy Equipment

This apparatus is heavy.

- The apparatus should be placed in a location that is sufficiently strong to support its weight (approximately 450 kg).
- The frame includes cross - members so that a fork lift, pallet truck or other lifting device can be used to transport it in an upright position.
- Where manual lifting is necessary, two or more people may be required for safety, and all should be made aware of safe lifting techniques to avoid strained backs, crushed toes, and similar injuries.
- Safety shoes and/or gloves should be worn when appropriate.

Chemical Safety

Operation of this equipment involves the use of toxic substances. Details of the chemicals intended for use with this equipment are given in the Operation section. Chemicals purchased by the user are normally supplied with a COSHH data sheet which provides information on safe handling, health and safety and other issues. It is important that these guidelines are adhered to.

- It is the user's responsibility to handle chemicals safely.
- Prepare chemicals and operate the equipment in well ventilated areas
- Only use chemicals specified in the equipment manuals and in the concentrations recommended
- Follow local regulations regarding chemical storage and disposal

Water Borne Hazards

The equipment described in this instruction manual involves the use of water, which under certain conditions can create a health hazard due to infection by harmful micro-organisms.

For example, the microscopic bacterium called Legionella pneumophila will feed on any scale, rust, algae or sludge in water and will breed rapidly if the temperature of water is between 20 and 45°C. Any water containing this bacterium which is sprayed or splashed

creating air-borne droplets can produce a form of pneumonia called Legionnaires Disease which is potentially fatal.

Legionella is not the only harmful micro-organism which can infect water, but it serves as a useful example of the need for cleanliness.

Under the COSHH regulations, the following precautions must be observed:-

- Any water contained within the product must not be allowed to stagnate, i.e. the water must be changed regularly.
- Any rust, sludge, scale or algae on which micro-organisms can feed must be removed regularly, i.e. the equipment must be cleaned regularly.
- Where practicable the water should be maintained at a temperature below 20°C. If this is not practicable then the water should be disinfected if it is safe and appropriate to do so. Note that other hazards may exist in the handling of biocides used to disinfect the water.
- A scheme should be prepared for preventing or controlling the risk incorporating all of the actions listed above.

Further details on preventing infection are contained in the publication "The Control of Legionellosis including Legionnaires Disease" - Health and Safety Series booklet HS (G) 70.

CONTINUOUS DISTILLATION COLUMN
UOP3CC

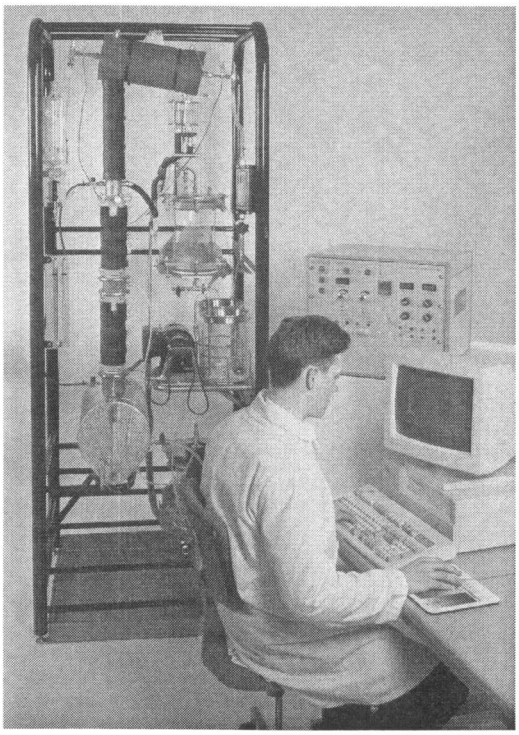
Contents

1 Introduction to the Equipment

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

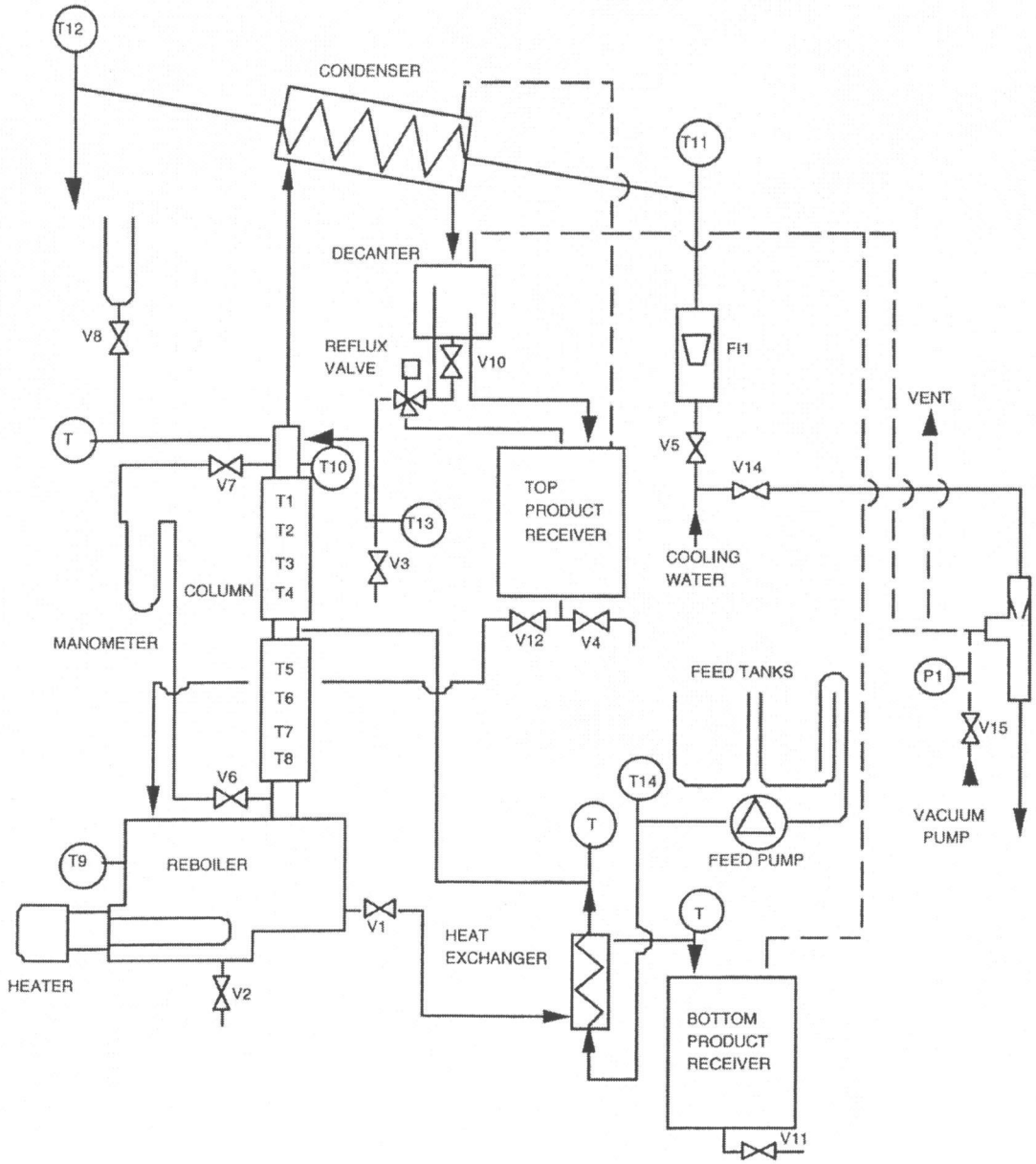
Laboratory scale distillation columns are needed to provide adequate practical training for student engineers and plant operators in a safe environment. They may also be used to acquire process separation data, of use in full-scale plant design.

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

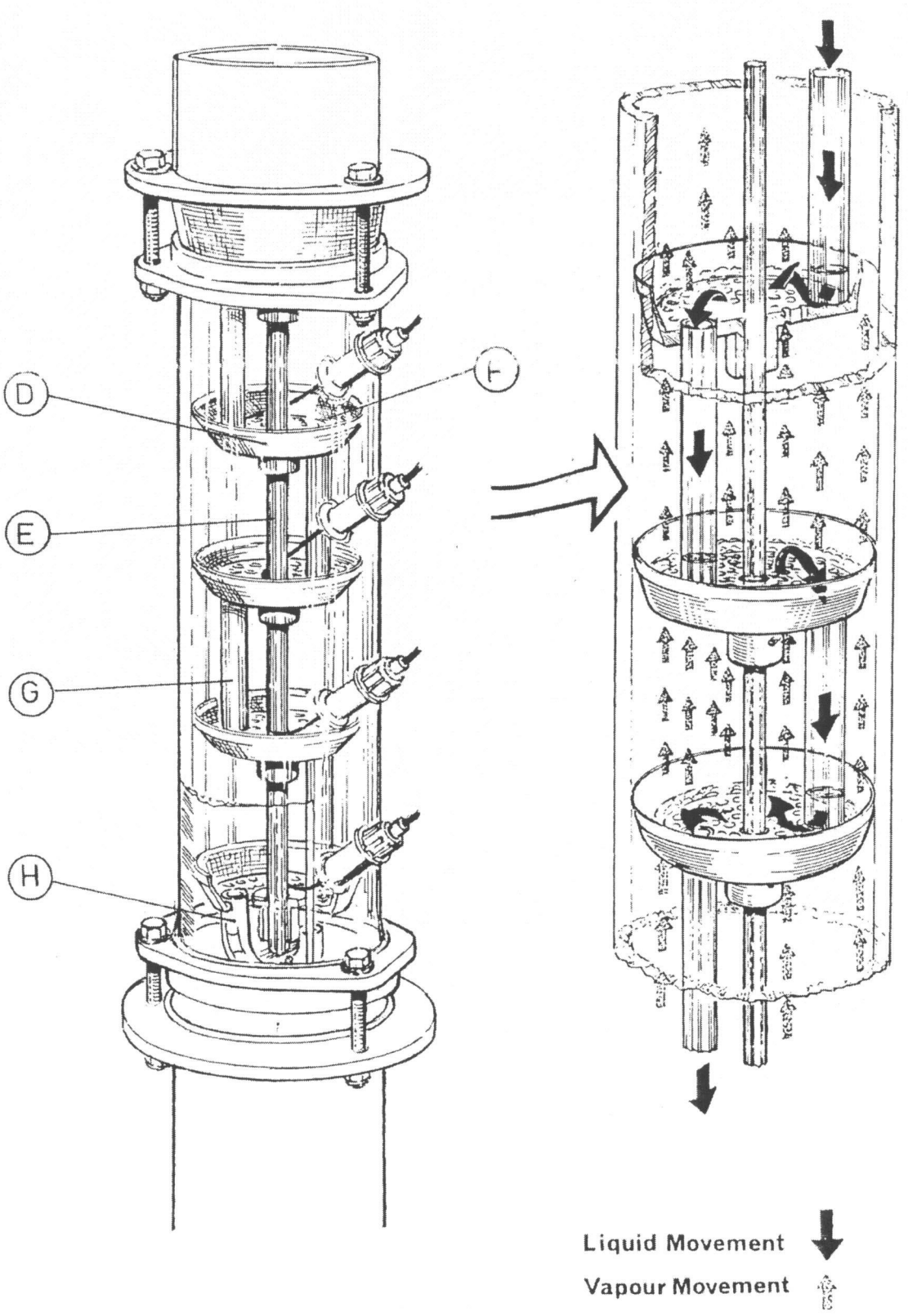


UOP3CC Continuous Distillation Column
with computer control

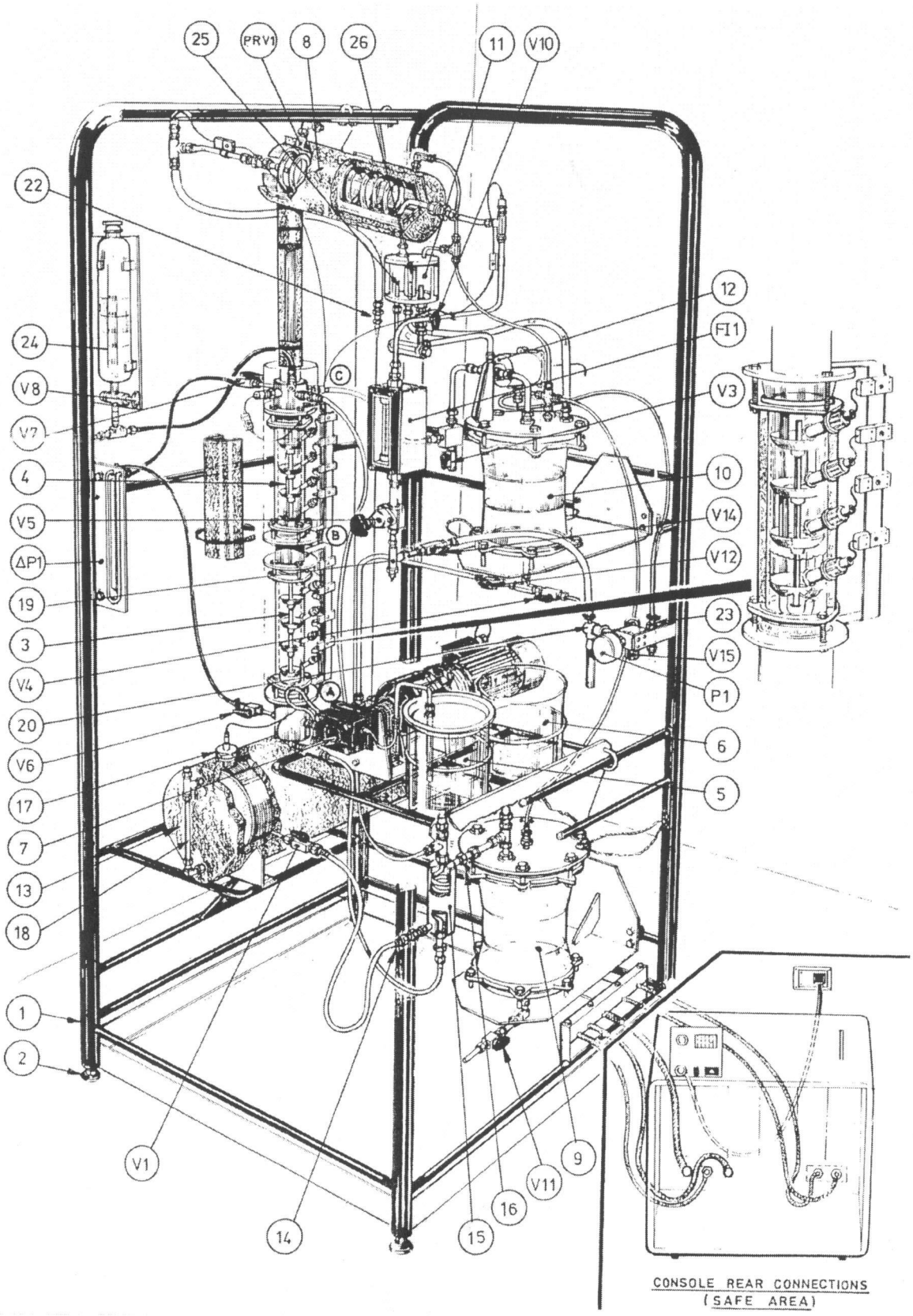
1.1 Schematic Diagram of Apparatus



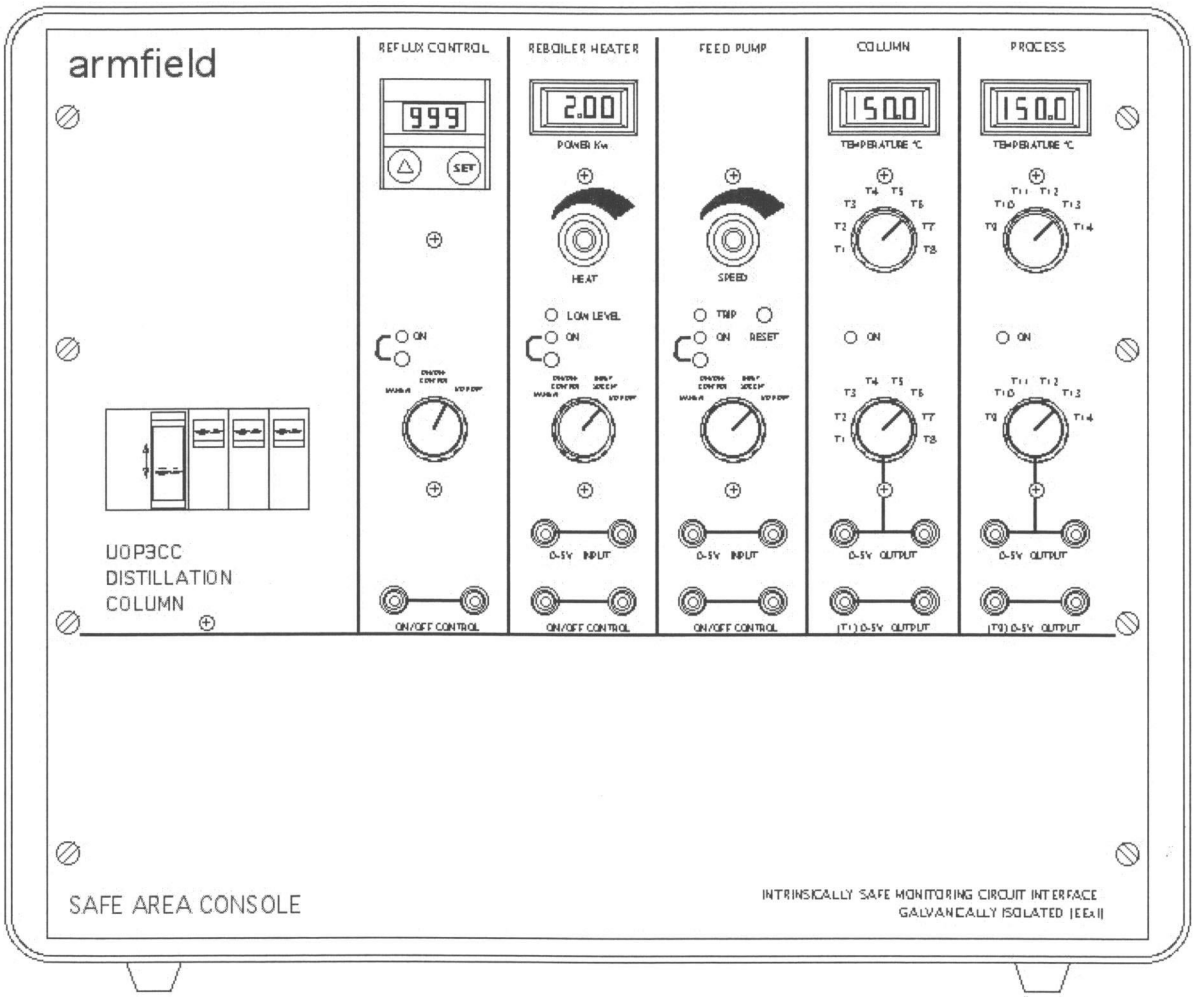
1.2 Diagram of Distillation Column



1.3 Diagram of UOP3CC process unit



1.4 Diagram of UOP3CC Console



2 Description

Where necessary, refer to the drawings on pages 2 to 5.

2.1 Overview

The UOP3CCContinuous Distillation Column is a self-contained distillation facility consisting of two interconnected units: a floor standing process unit and a bench-mounted control console.

2.2 Floor-Standing Frame

The distillation column is mounted on a floor standing, welded tubular steel framework (1) fitted with four adjustable feet (2). The frame is designed to allow the use of a fork lift or pallet truck to manoeuvre the unit into position initially.

2.3 Distillation Column

The 50mm diameter sieve plate column is made up of two glass sections (3) and (4) each containing four sieve plates. The columns are separated by a central feed section and arranged vertically for counter-current vapour/liquid flow. The column is insulated to minimise heat loss.

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

Feed mixture from either of the feed tanks is pumped by pump (7) to the base, centre or top of the distillation column at connections (A), (B) or (C) respectively. The feed pump incorporates a length of Viton rubber tubing. This tubing is suitable for all of the recommended test mixtures (see "Operational Procedures"). Where other test mixtures are being used, the suitability of this material must be checked.

A packed column is supplied loose. This column can be fitted in place of the sieve plate column where it is required to demonstrate the characteristics of a packed column.

Note: The packed column does not incorporate temperature sensors. The control experiments using an external PID controller or PLC and the control/data logging software cannot be performed when this column is fitted.

2.4 Reboiler

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

In continuous operation, valve (V1) is open and bottom product flows from the reboiler through the bottom product cooler (15) to the bottom product tank (9). It is possible to preheat the feed to the column by directing the feed through a spiral coil in

the bottom product cooler where heat is transferred from product leaving the reboiler at the boiling point. When feeding cold feed directly to the column, the product from the reboiler is cooled in the bottom product cooler by circulating cold water through the spiral coil.

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

The column and reboiler are both insulated to minimise heat loss. A level sensor (17) inside the reboiler protects the heating element from overheating due to low operating level and a sight glass (18) allows the level in the reboiler to be observed.

2.5 Condenser

Vapour from the top of the column passes to a water-cooled, coil-in-shell condenser (8), which may be fitted with an insulated jacket to allow heat balances to be carried out. (The insulated jacket should not be fitted to the condenser for normal operation.) The shell of the condenser incorporates a pressure relief valve (PRV1) to protect the system in the event of a blocked vent and cooling water failure. Cooling water enters the condenser at a regulated rate through a variable area flowmeter (FI1) and the flowrate is controlled by diaphragm valve (V5). A cooling water supply is connected to the inlet nozzle (19) and serves also to operate the vacuum pump (20) when operation at reduced pressure is required. Water supply to the vacuum pump is controlled by valve (V14), which must only be operated when valve (V5) is open.

2.6 Decanter

Condensate is collected in a glass decanter (11) (phase separator) which is by-passed for normal distillation experiments by opening valve (V10). When the decanter is in use (separation of two immiscible liquids as condensate), valve (V10) is closed so that the overflow (25) and underflow (26) pipes inside the vessel, can take effect.

With valve (V10) open, condensate from the condenser outlet passes directly through the decanter to the inlet of the reflux ratio control valve (12) which is a 3-way solenoid operated valve. Depending on the setting of the reflux timers, condensate is directed by the reflux valve either back to the top of the column or to the top product collecting vessel (10). When directed to the column, the reflux passes through a U-seal where a valve (V3) can be used for measuring boil-up rate or for draining the U-seal. The contents of the top product tank (10) can be drained into the reboiler (13) for re-use via valve (V12).

2.7 Thermocouples

Temperatures within the system are monitored by fourteen thermocouple sensors (T1 to T14) located at strategic positions in the system. T1 to T8 are located in the column and measure the temperature of the liquid on each sieve plate. There are seventeen locations for the temperature sensors, three of which do not have sensors installed but which can be fitted with sensors moved from other, less relevant locations when necessary.

2.8 Manometer

The total pressure drop across the column is indicated on a U-tube manometer (ΔP1) via appropriate tappings in the column fitted with isolating valves (V6) and (V7).

2.9 Product Receiver

All of the vessels in the system are connected to a common vent on the top product receiver. This vent is normally connected through a 4.0m length of tubing to a fume cupboard or safe atmospheric vent outlet.

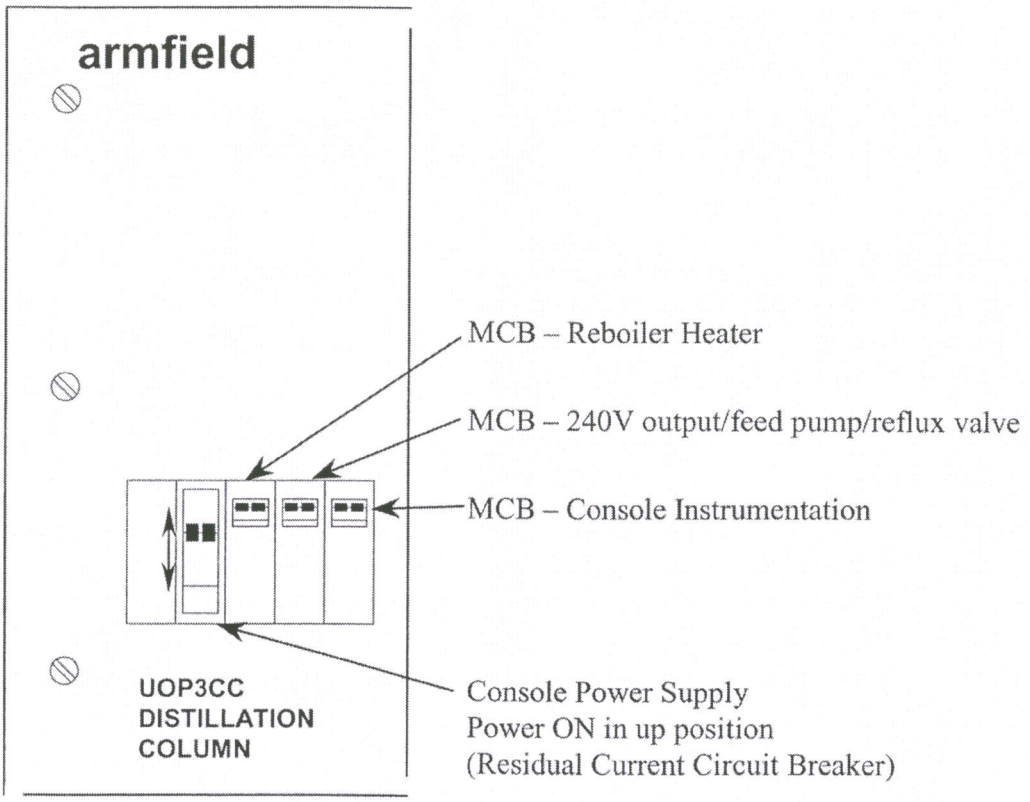
2.10 Vacuum Pump

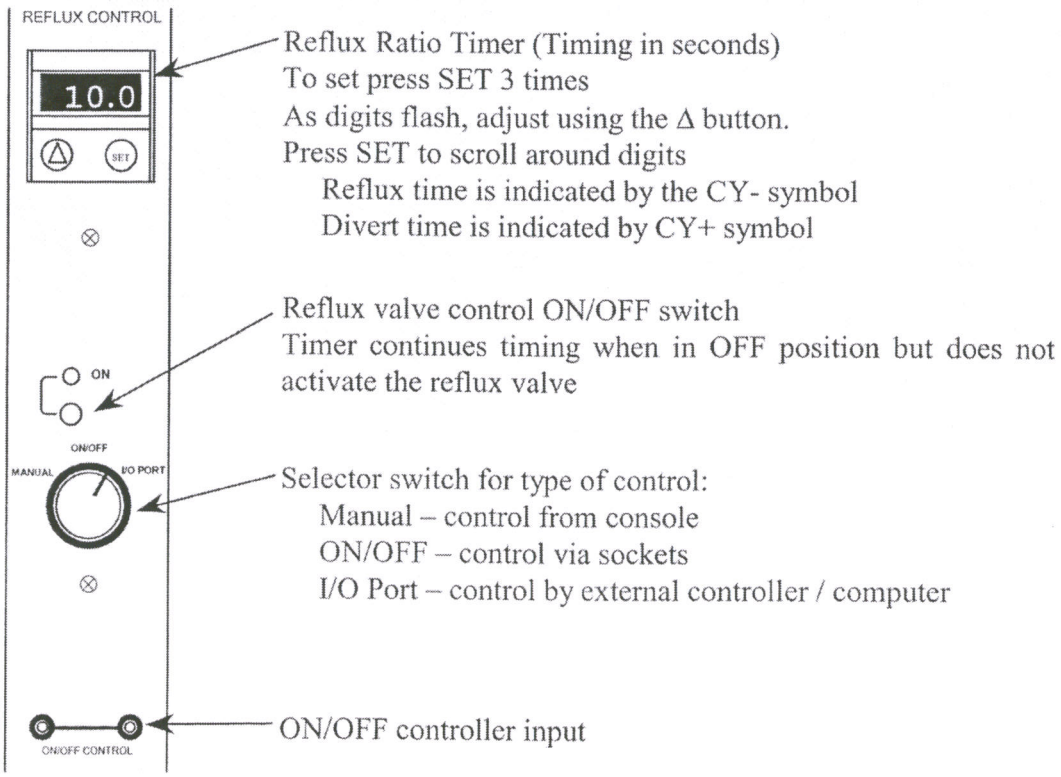
Operation at reduced system pressures is achieved using the water powered vacuum pump (20). When in use, the flexible vent pipe from the common connection on the top product receiver is attached to the inlet of this vacuum pump at (23), and motive water admitted via valve (V14), which must only be operated when valve (V5) is open. The level of vacuum is adjusted using needle valve (V15) and is indicated on pressure gauge (P1).

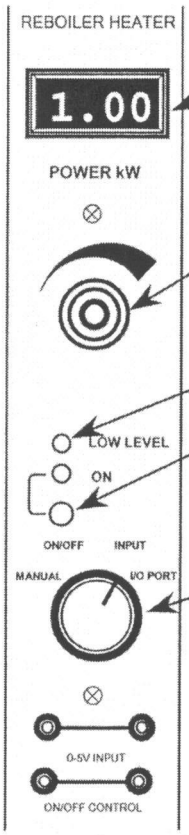
2.11 Control Console

The console is attached to the process unit by an umbilical cable which is of adequate length to allow the console to be positioned at least 2.0m away (outside the "Zone 2" area). See the Safety section at the front of this manual, and the Specifications section on page 22.

The following pages provide a description of the console controls and connections.


armfield
MCB – Reboiler Heater
MCB – 240V output/feed pump/reflux valve
MCB – Console Instrumentation
UOP3CC
DISTILLATION
COLUMN
Console Power Supply
Power ON in up position
(Residual Current Circuit Breaker)


REFLUX CONTROL
10.0
SET
Reflux Ratio Timer (Timing in seconds)
To set press SET 3 times
As digits flash, adjust using the Δ button.
Press SET to scroll around digits
Reflux time is indicated by the CY- symbol
Divert time is indicated by CY+ symbol
ON
Reflux valve control ON/OFF switch
Timer continues timing when in OFF position but does not activate the reflux valve
ON/OFF
MANUAL
I/O PORT
Selector switch for type of control:
Manual – control from console
ON/OFF – control via sockets
I/O Port – control by external controller / computer
ON/OFF CONTROL
ON/OFF controller input



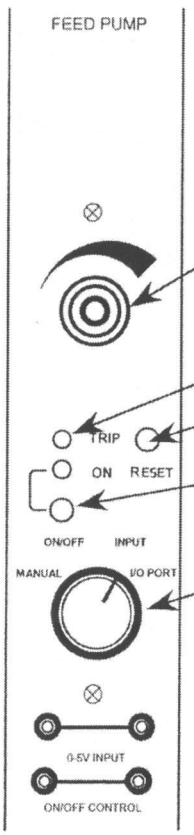
Power input to the heating element

Manual power adjustment for the heating element

Reboiler low level indicator (light on when low)

Reboiler heater control ON/OFF

Control selector switch:
- Manual – control from console
- ON/OFF – external control via ON/OFF sockets
- Input – external control via 0-5V sockets
- I/O Port – Control by external controller / computer



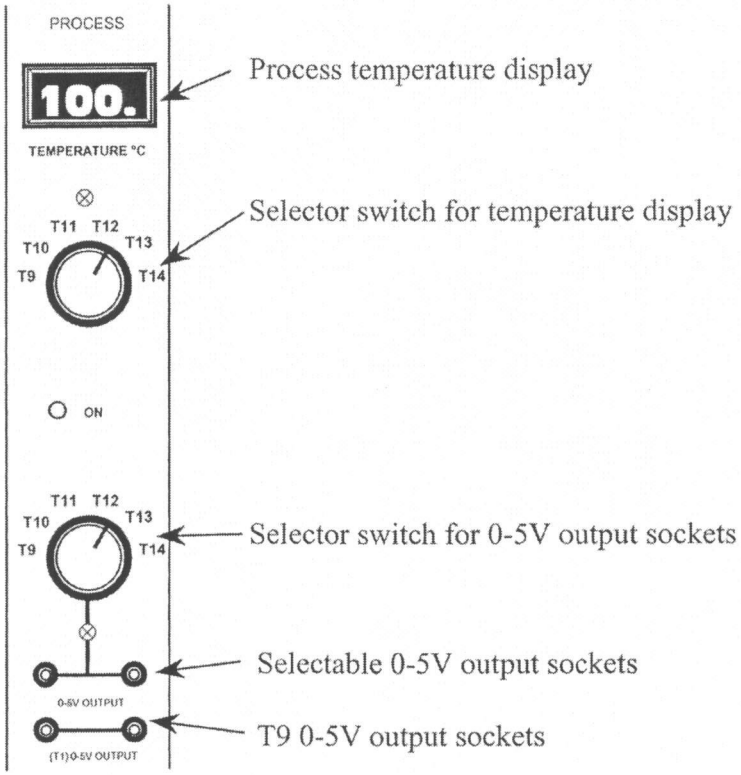
Manual adjustment of feed pump speed

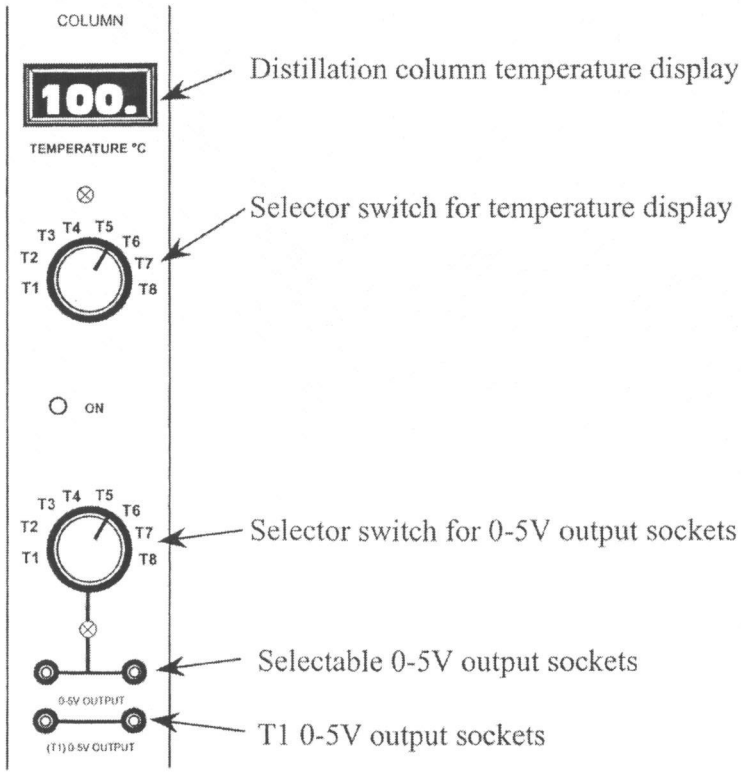
Motor fault indicator (light on when fault)

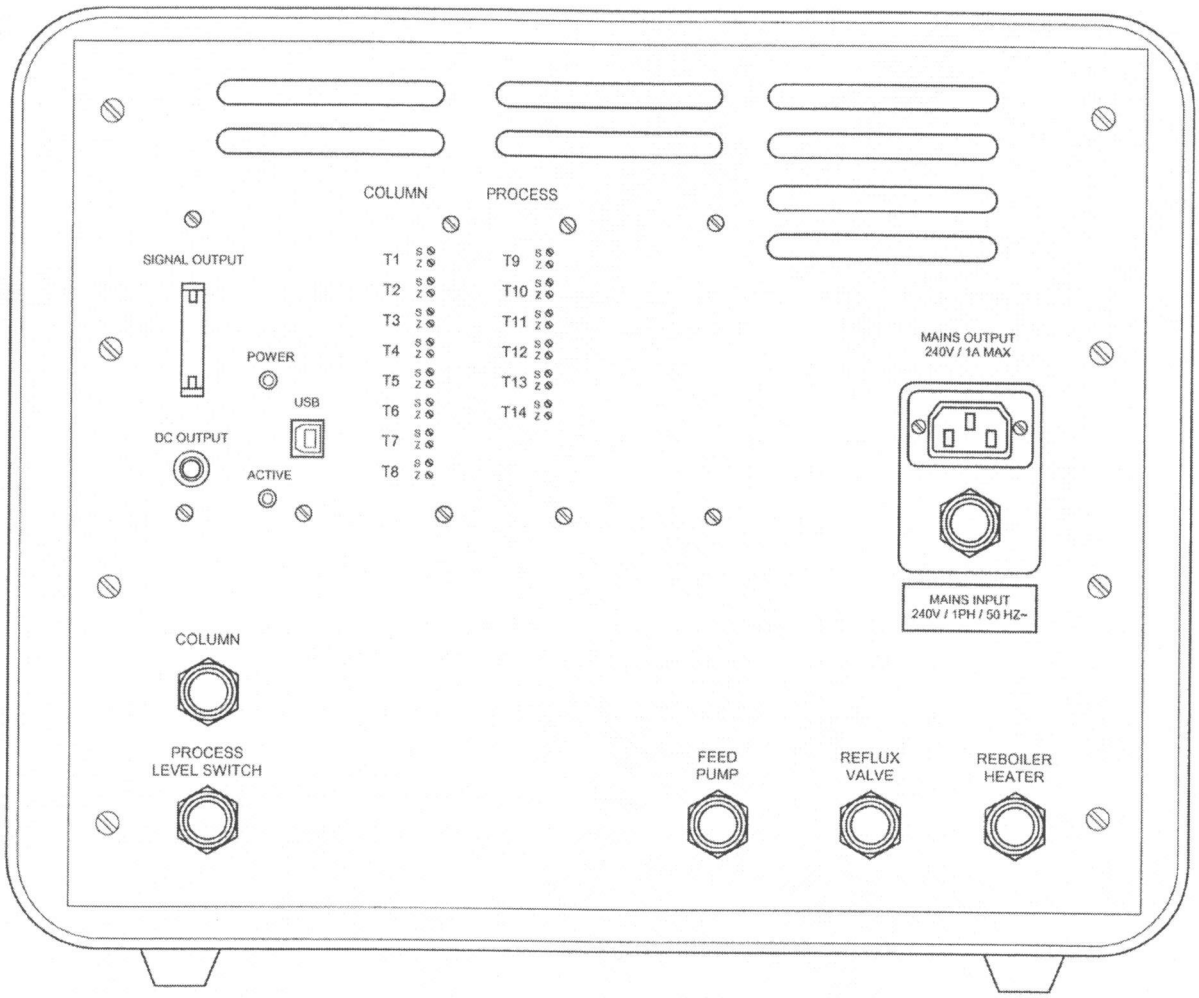
Motor reset

Feed pump motor ON/OFF

Control selector switch:
- Manual – control from console
- ON/OFF – external control via ON/OFF sockets
- Input – external control via 0-5V sockets
- I/O Port – Control by external controller / computer


PROCESS
100.
TEMPERATURE °C
T11 T12
T10 T13
T9 T14
ON
T11 T12
T10 T13
T9 T14
0-5V OUTPUT
(T1)0-5V OUTPUT
Process temperature display
Selector switch for temperature display
Selector switch for 0-5V output sockets
Selectable 0-5V output sockets
T9 0-5V output sockets


COLUMN
100.
TEMPERATURE °C
T3 T4 T5
T2 T6
T1 T7 T8
ON
T3 T4 T5
T2 T6
T1 T7 T8
0-5V OUTPUT
(T1) 0-5V OUTPUT
Distillation column temperature display
Selector switch for temperature display
Selector switch for 0-5V output sockets
Selectable 0-5V output sockets
T1 0-5V output sockets



The rear of the UOP3CC Console houses all cable connection glands, mains connections and DC connections.

Two data ports are provided, a USB port for direct connection to a PC with Armfield software, and a 20-way signal output port, which provides voltage outputs for each of the sensor readings.

The rear panel also houses zero and span potentiometers for each of the thermocouples, in order that the displays on the front of the console can be adjusted to read correctly.

3 Operation

Where necessary, refer to the drawings on pages 2 to 5 and to the console description beginning on page 8.

3.1 Warning!

The vacuum pump must never be started before opening valve (V5) to allow cooling water to the condenser (8). Failure to observe this will cause solvent to be discharged to the drain with the vacuum pump motive water.

3.2 Changing the Column

The process unit is provided with both a sieve plate column and a packed column.

To change from the sieve plate column to the packed column:

Disconnect the thermocouple sensors on the column from the connectors on the metal support bracket. Leave the sensors attached to the column.

If the central feed distributor has been in use, remove and flexible tubing attached to the distributor.

Undo and remove the top and bottom fixing bolts securing the thermocouple support bracket. Remove the bracket and place in a safe place. Retain all bolts removed during this procedure.

Undo and remove the fixing bolts from the top and bottom flanges on the top glass column section. Remove the top column section. If necessary to assist in removing the top section, there is a sliding flange positioned above the section which may be moved upwards towards the condenser.

Place the top column section in a safe location. Retain the PTFE sealing ring.

Undo and remove the fixing bolts at the top of the bottom glass column section. Remove the central feed redistributor and place in a safe place.

Undo and remove fixing bolts at the bottom of the lower glass column section. Remove the column section and place in a safe location. Retain the PTFE sealing ring.

Position a PTFE sealing ring at top and bottom of the packed column and place the packed column in position. Slide down the upper sliding flange on to the top of the column.

Secure the top and bottom of the column with fixing bolts. Tighten the bolts gradually in sequence until the column is well seated, but do not over-tighten as this will damage the sealing rings.

To change from the packed column the sieve plate column:

Undo and remove the fixing bolts from the column. Retain all bolts from this procedure. Additional bolts will also be required (which should have been retained when the sieve plate column was previously removed).

Remove the packed column, retaining the PTFE sealing rings.

Both glass sieve plate column sections are physically identical, but may have different thermocouple sensor labels. The lower column section 5 to T8. Place a PTFE sealing ring on the bottom of this section and place the section onto the bottom flange. Secure the bottom section with fixing bolts. Tighten the bolts gradually in sequence until the column is well seated, but do not overtighten as this will damage the sealing rings.

Position the central feed redistributor on the top of the bottom column section and secure with fixing bolts.

Position a PTFE sealing ring on the top of the top column section and place the section on top of the feed redistributor. The sliding flange above the top section may be moved upwards to assist in positioning the column section.

Move the sliding flange back down snugly on top of the top column section and secure the top section to the feed distributor and the top flange using fixing bolts. Tighten the top bolts gradually in sequence until the column is well seated, but do not over-tighten as this will damage the sealing rings.

Fit the metal thermocouple support bracket and secure it with bolts at top and bottom.

Connect the thermocouples to the support bracket.

If the central feed redistributor is to be used, reconnect the flexible tubing to the redistributor.

3.3 Reflux Ratio Control

The reflux ratio timer on the control console is used to set the quantity and frequency of condensate returning to the distillation column. With the timer switched off, all of the condensate will be directed to the column (total reflux).

3.3.1 Typical reflux ratio examples

If the reflux ratio required is 2:1 and the total cycle time required is 21 seconds:

Condensate will be directed by the reflux ratio valve to the column for 14 seconds then to the top product receiver for 7 seconds. This cycle will then be repeated continuously until different values are inserted to the controller or until the reflux control is switched off.

If a ratio of 4:1 is required over the same cycle time:

Condensate will be directed to the column for 16.8 seconds and to the top product receiver for 4.2 seconds. The calculation is as follows:

$4 + 1 = 5$; $21/5 = 4.2$; $4 \times 4.2 = 16.8$; $(21 - 16.8 = 4.2)$

3.3.2 Setting the controller

The time range and mode of the controller can only be set when the electrical supply to the controller from the control console is switched off.

Switch off the reflux controller switch on the control console. This switches off the power supply to the controller. The controller is now not controlling reflux flow. Because the controller has an internal battery, the display is still illuminated and the controller settings may be adjusted.

Press the SET button on the reflux ratio timer.

The controller should be set to Immediate Cycle (CY) mode. If CY is not already set, use the Δ button to cycle through the modes until CY is displayed.

Press the SET key again.

Select the time range required. Seconds x 10 is suggested as the most suitable time range, allowing cycle times of between 00.1 and 99.9 seconds to be set. Use the Δ button to cycle through the modes until secx10 is displayed.

The set time may then be adjusted at any time (even when the controller is switched on), as follows (this assumes that secx10 mode has been selected):

Set the time interval during which condensate should be directed to the top product receiver (CY+ on the controller):

Press SET to select the 10 digit

Press Δ to set the 10 digit

Press SET to select the 1 digit

Press Δ to set the 1 digit

Press SET to select 0.1 digit

Press Δ to set the 0.1 digit

Set the time interval during which condensate should be directed back to the column (CY- on the controller):

Press SET to select the 10 digit

Press Δ to set the 10 digit

Press SET to select the 1 digit

Press Δ to set the 1 digit

Press SET to select 0.1 digit

Press Δ to set the 0.1 digit

Press SET to end time adjustment. To begin controller operation, switch on power to the reflux controller using the reflux controller on/off switch.

The CY- digit in the bottom right of the display now indicates flow of condensate back to the column. The CY+ digit indicates flow of condensate to the top product tank.

To display the set time during operation, press SET once to display set time CY+, twice to display set time CY-.

3.4 Measuring Temperatures

There are thirteen temperature sensing stations on the equipment, which are designated as follows:

T1	=	Top tray of distillation column
T2	=	2nd tray
T3	=	3rd tray
T4	=	4th tray
T5	=	5th tray
T6	=	6th tray
T7	=	7th tray
T8	=	8th tray
T9	=	Temp of liquid in reboiler
T10	=	Temp of vapour leaving the column above tray 1
T11	=	Temp of cooling water entering condenser
T12	=	Temp of cooling water leaving condenser
T13	=	Temp of condensate as reflux/top product
T14	=	Temp of feed liquid from feed tank

It is intended that both thermocouples T11 and T12, can be moved to any of the three positions marked T on the flow diagram. This will be necessary when carrying out a feed preheat experiment or an azeotropic distillation experiment.

In fact all of the thermocouples are identical so any can be moved to different locations but T11 and T12 are the recommended "movable" sensors as their connecting cables will not require any special re-routing.

When moving sensors, always ensure that the blank fitting removed from the new sensor location is used to blank off the fitting from which the sensor was removed.

Temperatures may be viewed on the display on the right-hand panel of the control console. To display any temperature from T1 to T8, set the upper selector dial to the corresponding station designation. To display temperatures T9 to T14, turn the upper selector dial fully clockwise (to the furthest right-hand setting) and then set the lower selector dial to the station designation required.

3.5 Measuring Column Pressure Drop

The overall pressure drop over the column can be measured using the manometer $\Delta P1$.

Always open V6 before V7, take the pressure reading then immediately close both valves. This will reduce the risk of contamination of the manometer water by the hydrocarbons.

Also to prevent contamination, never open valves V6 or V7 when flooding is occurring on the sieve plates (boil-up rate too high).

3.6 Taking Samples for Analysis

Samples for analysis can be taken from pertinent points in the system as follows:-

Feed liquid	- From feed tank
Liquid in reboiler	- V2 (**WARNING!** Liquid at boiling point!)
Condensate from condenser	- V3 (reflux/top product)
Top product receiver	- V4
Bottom product receiver	- V11

Note: When using valve V3 to obtain a sample of top product or to measure boil up rate the valve should **not** be fully opened, to prevent vapour from escaping. Gradually open valve V3 until flow of reflux into the column stops but liquid is retained in the flexible connecting pipe. Small adjustments of the valve position can be applied to maintain the desired level in the pipe. Provided that the same level in the pipe is maintained at the start and finish of the timing operation then the boil up rate measured will be accurate.

3.7 Feed pump calibration

The peristaltic feed pump is designed to give approximately 1 ml/min per revolution of the drive shaft. As the variable speed motor is capable of speeds varying from 0 to 300 RPM, the pump will be able to deliver approximately 0 to 300 ml/min of feed to the column.

In order to achieve greater accuracy, it is necessary to produce a calibration graph of the actual flowrate against the position of the variable speed dial on the control console. The dial has ten full turns, each full turn marked in one hundredth segments. To produce the graph it will only be necessary to measure the flow at ten settings over the full range.

Disconnect the feed tubing to the distillation column. Ensure the feed tank with the pump suction pipe inserted has sufficient water for the calibration. Using a 250 ml graduated cylinder and a stop watch, simply determine the flowrate at the ten settings and construct a graph which can be subsequently located near the control console.

If the unit is to be controlled via a PC, the calibration curve for the feed pump can be created and stored within the Armfield software (see the software help for more details).

Note: The distillation column has been designed for feed rates between 50 and 200 ml/min depending on the chemicals being used so a slight inaccuracy at the minimum and maximum settings of the pump speed will not affect the process.

3.8 Suggested Test Mixtures

The following binary mixtures have been selected as the most suitable for use with the distillation column.

Methyl*cyclo*hexane/Toluene

Toluene/Chlorobenzene

Cyclohexane/*n*-Heptane

Methanol/Water

Chlorobenzene/Ethylbenzene

Methanol/Ethanol

Ethanol/Water

The equilibrium data of these systems is well tabulated and can be found in the booklet

"Recommended Test Mixtures For Distillation Columns",

Ulfert Onken and Wolfgang Arlt,

Issued by The Institution of Chemical Engineers,

165-171 Railway Terrace, Rugby, Warwickshire, CV21 3HQ, England

Tests carried out by Armfield Ltd have utilised the systems Methyl*cyclo*hexane/Toluene and a mixture for use in a non flameproof environment, Trichloroethylene/Methylene Chloride*.

It is important that data is available for liquids used in the distillation column so their suitability can be checked, for example:-

	B. Point °C	Flash Point °C	Ignition Temp. °C	T Class	Apparatus Group
Toluene	111	6	535	T1	IIA
Ethanol	78	12	425	T2	IIA
Cyclohexane	81	-18	259	T3	IIA
Methylcyclohexane	101	-4	260	T3	IIA
Chlorobenzene	132	28	637	T1	IIA
n - Heptane	98	-4	215	T3	IIA
Methanol	65	11	455	T1	IIA
Ethylbenzene	136	15	431	T2	IIA

Many other liquids can be used but the information above is required to ensure that their characteristics are within the flameproof specification of the equipment which is:-

EEx d Group IIB Temp class T4

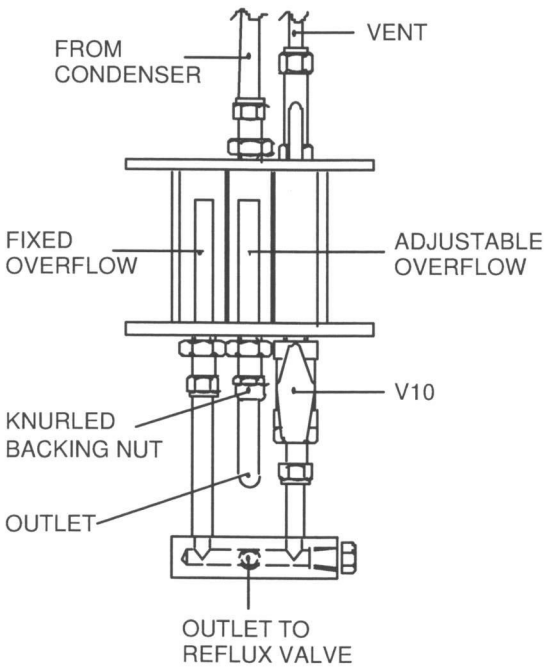
*It should be noted that most substances suitable for use in distillation are potentially hazardous, so while this mixture is 'safe' for use in a non flameproof environment it should **not** be considered harmless. All appropriate safety precautions must be taken and all relevant regulations must be followed during preparation for use, during operation of the equipment, and during the disposal of materials after use.

WARNING

When operating the column at reduced pressure, using the vacuum pump, it is essential that vapour is not drawn from the vent system into the water flowing to drain.

This can be avoided by careful choice of the binary mixture used in conjunction with the ambient conditions (air and cooling water temperatures). If vapour is observed in the clear vent pipework then the vacuum system should be turned off by closing valve V5.

3.9 Operation of the Decanter (Phase Separator)



The decanter in normal operation is used with valve (V10) open which allows condensate entering the vessel to flow directly to the reflux valve.

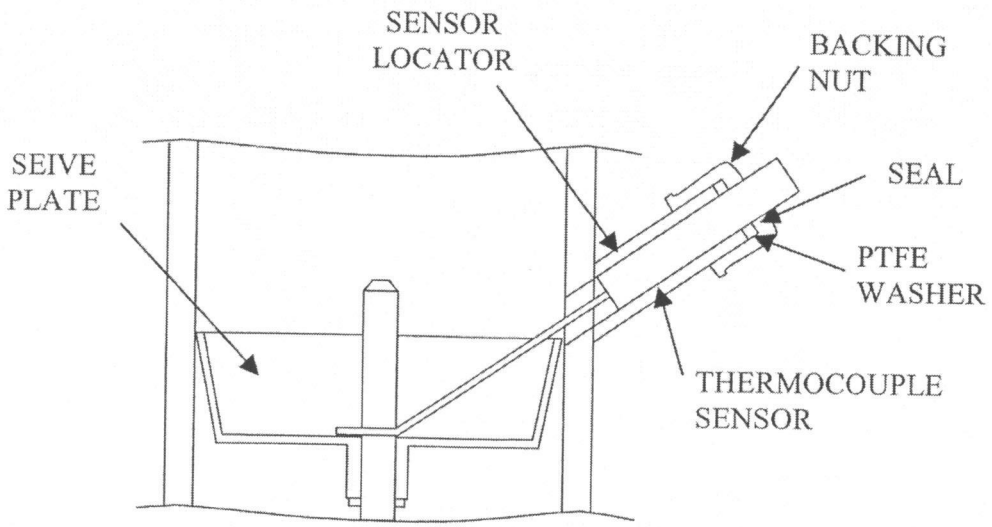
When carrying out an experiment which utilises a third liquid component, valve (V10) is closed and the decanter comes into operation. The condensate entering the decanter will be made up of the miscible binary mixture plus an immiscible component. The heavier component will separate and collect at the base of the decanter and its' level will begin to rise. Eventually the lighter phase will overflow the fixed overflow and, when the level is sufficiently high, the heavier phase will overflow the adjustable overflow. The adjustable overflow will always be below the level of the fixed

overflow and when adjusted will determine the height of the interface between the light and heavy components.

As a guide, begin the process with the adjustable overflow 1cm below the level of the fixed overflow.

3.10 Adjustment of feed position

Feed to the distillation column from the feed pump (7) will normally be to the top, centre or base of the column. When the flexible pipe carrying the feed is connected to one of these positions the other two positions are fitted with blanking caps. It is also possible to connect the feed to any of the sieve plates by removing the relevant thermocouple and replacing with the feed pipe. To do this, first unplug the yellow thermocouple sensor connector, then release the plastic backing nut. Remove the PTFE washer and seal from the sensor and, after positioning the backing nut on the pipe, insert the washer and seal. Push the end of the pipe into the sensor locator to the desired position and carefully tighten the backing nut.



3.11 Operation of the Reboiler

Heating of the liquid in the reboiler is achieved by an electrical heating element. The maximum power of the element is 2.0 kW and this is adjustable at the control console.

Due to the various flux requirements of the liquids which can be used in the reboiler, the heater must always be switched on at zero power (adjustment fully anti-clockwise). The power can then be increased carefully until boiling is achieved and fine adjustment is carried out to cause the required activity on the sieve plates (observed on Trays 1 and 5). Excessive power to the reboiler may cause vapour to escape from the vent pipe due to overloading of the condenser.

NOTE: The reboiler heater maximum power is 2.0 kW rated at a supply voltage of 240V (120V). 2.0 kW will not be achieved if the supply voltage is low but this will not affect the process as maximum power is rarely, if ever, required.

3.12 Using the UOP3CC with supplied software

The UOP3CC is supplied with educational software on CD-ROM. Ensure that the software is installed as described in the Installation Guide.

The PC with the

To run the software, open the 'Start' menu, and choose 'UOP3cc Distillation Column' from the 'Armfield Unit Operations Software' group. The initial screen will load displaying the first page of the presentation screen.

The toolbar at the top of the screen contains four buttons which are used to navigate the software:

- View Diagram – displays a mimic diagram of the apparatus, with sensor readings displayed in real time. Data values can be recorded by clicking the 'GO' button.
- View Graph – displays a graph of selected recorded values.
- View Table – displays a table of recorded data.
- View Presentation – displays the presentation screens.

Help texts are available within the software explaining how to use the software, and detailing experimental theory and procedures.

The software can be used with any of the Teaching Exercises listed in this manual to aid with data recording, and to provide online help for the student.

4 Specifications

4.1 Overall Dimensions (process module)

Height: - 2.25m

Width - 0.85m

Depth - 0.80m

4.2 Overall Dimensions (console)

Height: - 0.30m

Width - 0.33m

Depth - 0.40m

4.3 Electrical Supply

	UOP3CC-A	UOP3CC-B
Green/yellow lead	Earth (Ground)	Earth (Ground)
Brown lead	Live (Hot)	Live (Hot)
Blue lead	Neutral	Neutral
Fuse rating	10A	20A
Voltage	220-240V	110-120V
Frequency	50Hz	60Hz

4.4 Cold Water Supply

The equipment requires connection to a clean water supply with a pressure of 2 bar and a flow rate of 15 litres/minute.

The equipment must be connected to the water supply using 12mm ID flexible hose (not supplied).

In hot climates the cold water supply temperature may be too warm to completely condense the test mixture under evaluation. Vapour or condensate will be visible in the flexible vent pipework if either the cooling water flowrate is too low or the cooling water inlet temperature is too high. Under these circumstances a chilled water supply may be required. If the vacuum pump is not in use then this can be limited to 4.4 litres/minute at 2 bar pressure.

4.5 Connection to Drain

Water exiting the condenser should be directed to a suitable drain capable of accepting warm water at up to 15 litres/minute.

The equipment must be connected to drain using 12mm ID flexible hose (not supplied).

Water exiting the vacuum pump may contain traces of solvent. If local regulations do not allow discharge of water containing even small amounts of solvent to drain, then the water exiting the vacuum pump must be passed through a separator vessel to remove any solvent traces.

4.6 Solvent vapour extraction (ventilation requirements)

All solvent vessels and pipework are connected to a common vent pipe at the right-hand rear of the equipment (attached to the common connection on the top product receiver). It is recommended that, for operation under atmospheric pressure, the flexible PTFE tubing provided be connected to this and routed to a safe place outside the building or to a fume cupboard so that any vapours produced by abnormal conditions will be dispersed safely. For operation under reduced pressure conditions, the vent pipe must be connected directly to the inlet of the vacuum pump.

4.7 Bund (Spillage containment)

A stainless steel bund tray is provided which must be placed directly beneath the process unit on the floor, between the four support legs. This is designed to contain any accidental spillage within the confines of the framework and thus within the Zone 1 area.

4.8 Required specification for optional PC software

The UOP3CC does not require a computer for operation as control and sensor output measurement is provided by the control console. However, software is available if desired, and use of the software requires connection to a compatible Windows™ PC.

The software permits data-logging and control exercises to be carried out. It features a schematic representation of the apparatus, with data values displayed in real time, full data logging facilities and has both graphical and tabular displays of the recorded data.

To run the software, users must have access to a PC with the following minimum specification.

- Pentium processor or equivalent
- 16 MB RAM
- 10 MB hard disk space
- SVGA display (800 x 600 high colour)
- USB port
- CD-ROM drive
- MS Windows 98 / 2000 / XP

4.9 I/O Port connections

A 20-way data I/O port is fitted to the rear of the UOP3CC console, which allows users to pass the data from the distillation column to an alternative computer interface or external controller. The following table lists the signal connections to the I/O Port:

Pin 1	Tray 1 temperature	(T1)	0-150 °C
Pin 2	Tray 2 temperature	(T2)	0-150 °C
Pin 3	Tray 3 temperature	(T3)	0-150 °C
Pin 4	Tray 4 temperature	(T4)	0-150 °C
Pin 5	Tray 5 temperature	(T5)	0-150 °C
Pin 6	Tray 6 temperature	(T6)	0-150 °C
Pin 7	Tray 7 temperature	(T7)	0-150 °C
Pin 8	Tray 8 temperature	(T8)	0-150 °C
Pin 9	Reboiler temp.	(T9)	0-150 °C
Pin 10	Tops temp.	(T10)	0-150 °C
Pin 11	Cold water in temp	(T11)	0-150 °C
Pin 12	Cold water out temp	(T12)	0-150 °C
Pin 13	Reflux temperature	(T13)	0-150 °C
Pin 14	Feed temperature	(T14)	0-150 °C
Pin 15	Reboiler measured power	(F1)	0-300 RPM
Pin 16	Analog Ground		
Pin 17	Not Used		
Pin 18	Not Used		
Pin 19	Not Used		
Pin 20	Not Used		

4.10 USB channel numbers

The Armfield Windows™-compatible software allows data logging of the sensor outputs. However, users may prefer to write their own software for data logging, and for the convenience of those wishing to do so, Armfield has provided additional USB drivers allowing operation of the equipment via the USB socket on the UOP3CC console. The relevant channel numbers are as follows:-

Channel 0	Tray 1 temperature	(T1)	0-150 °C
Channel 1	Tray 2 temperature	(T2)	0-150 °C
Channel 2	Tray 3 temperature	(T3)	0-150 °C
Channel 3	Tray 4 temperature	(T4)	0-150 °C
Channel 4	Tray 5 temperature	(T5)	0-150 °C
Channel 5	Tray 6 temperature	(T6)	0-150 °C
Channel 6	Tray 7 temperature	(T7)	0-150 °C
Channel 7	Tray 8 temperature	(T8)	0-150 °C
Channel 8	Reboiler temp.	(T9)	0-150 °C
Channel 9	Tops temp.	(T10)	0-150 °C
Channel 10	Cold water in temp	(T11)	0-150 °C
Channel 11	Cold water out temp	(T12)	0-150 °C
Channel 12	Reflux temperature	(T13)	0-150 °C
Channel 13	Feed temperature	(T14)	0-150 °C
Channel 14	Reboiler measured power	(F1)	0-300 RPM
Channel 15	Power	(F1)	0-2.5 kW

5 Routine Maintenance

To preserve the life and efficient operation of the equipment it is important that the equipment is properly maintained. Regular maintenance of the equipment is the responsibility of the end user and must be performed by qualified personnel who understand the operation of the equipment.

5.1 General

Before every use, check the peristaltic pump tubing for splits or other damage and replace any damaged tubing. Regularly change the position of the tubing passing through the pump. The tube will retain its elasticity for many hours of use but there is sufficient length to allow the tube to be moved to other positions and hence reduce the wear at any single positon.

The peristaltic feed pump (7) has a pivoting clamp on top of the pumphead for fitting the flexible tubing. Ensure that the adjusters on either side of the pumphead have been set to match the diameter of tube. The tubing supplied has a bore of 3.2mm so the indicator must align with the 3.2 mark when the head is unclamped. To install or replace the flexible tubing pivot the clamp upwards and backwards to expose the rotor. Load the tubing into the slot between the rotor and pumphead then pivot the clamp forwards and downwards. The clamp will click shut.

After use, release the head of the peristaltic feed pump by pivoting the clamp on the top of the pumphead into the upright position. This will prolong the life of the tubing. It is essential to release the clamp when the equipment is not being used to prevent permanent deformation of the tube.

The equipment should be disconnected from the electrical supply when not in use.

When not in use for an extended period, all chemicals should be drained from the equipment and the equipment purged of residual vapours.

Water should be drained from the equipment before storage.

5.2 RCD test

Test the RCD by pressing the TEST button at least once a month. If the RCD button does not trip when the Test button is pressed then the equipment must not be used and should be checked by a competent electrician.

5.3 Relief valve test

At intervals of six to twelve months (more frequently for particularly harsh environments) the relief valve must be checked to ensure that it has not stuck or seized. The valve is located on the top of the condenser unit. Remove the attached pipework and visually inspect the inside of the fitting for any sign of corrosion or blockage, ensuring that the valve ball is free to move. Reassemble the fitting after checking the valve.

5.4 Earth test

The most critical safety component in any barrier installation is earthing. At intervals of not more that two years (more frequently for particularly harsh environments) the earth system of barrier installation should be examined and tested as follows:-

- Visually examine the earth conductors and ensure that they are not damaged in any way and that their terminations are secure and free from corrosion.
- Using a low-volt, low current test meter (i.e. a meter whose output does not exceed 30V and 50mA) measure the resistance between the earth busbar and the neutral star point of the supply and ensure that it does not exceed 1Ω. Record the reading. A consistent reading repeated over a long period of time is indicative that the earth return is sound and likely to remain so.

5.5 Routine inspection (including wiring diagrams)

At intervals of not more than two years (more frequently for particularly harsh environments) visually check the barrier installation and ensure that:-

- Barriers are of the types and polarities specified in the safety documentation.
- The barriers are securely attached to the earth busbar, thus making a good connection to the IS earth. Use a torque wrench or spanner to check the correct tightness of the two M4 self-locking nuts that secure each barrier to the busbar. The recommended torque is 2.3Nm (20.3 lb in.).
- There are no signs of damage or corrosion to the barriers or the IS earthing system.
- All connections are properly made. The tightness of the hazardous-area and safe-area terminals on barriers should be checked with a torque screwdriver having a 3.5mm blade width. The torque setting should be approximately 0.4Nm (3.5 lb in.).
- Interconnecting cables are of the type and rating specified in the safety documentation, are correctly routed and segregated, and are not frayed or otherwise damaged.
- All earth returns and cable screens from the hazardous-area are properly connected to earth via an earth rail and earth terminals. On 1-channel barriers the earth and returns and cable screens may be connected via terminal 4 of the barrier with which they are associated because that terminal is internally connected to the two earth studs.
- The bearings show no signs of overheating, and make no abnormal noise during operation.

In addition the peristaltic pump tubing should be completely replaced, even if there are no obvious visual signs of wear or damage.

5.6 Inverter Settings

The inverter parameters are set prior despatch and should not require modification under normal circumstances.

The following procedure is included to allow the settings to be corrected if the original parameters become corrupted. The majority of the inverter parameters remain set to the manufacturers default value and only settings that differ from the default are listed below.

The inverter is located in the bottom of the electrical console and can be accessed by turning the console upside down and removing the bottom cover.

Press PRG/RESET key, **0.Fnc** should be displayed.

Press ∧ key until **1.F__** is displayed.

Press FUNC/DATA key.

Press ∧ key until F 01 is displayed.

Press FUNC/DATA key.

Press ∧ or ∨ key to set the required value for F 01.

Press FUNC/DATA key to save the value and display the next parameter.

Set the parameters listed below then press PRG/RESET to return to normal operation.

F01	**1**	**(Speed set by external signal)**
F02	**1**	**(Operation by FWD signal)**
F03	**55**	**(Max frequency)**
F11	**2.0**	**(Electronic thermal overload setting A)**
F37	**2**	**(Auto torque boost)**
P01	**2**	**(No of poles)**
P02	**0.18**	**(Motor power kW)**
P03	**1.3**	**(Full load current A)**
P06	**0.63**	**(No load current)**
P07	**8.68**	**(Motor characteristic R1)**
P08	**13.54**	**(Motor characteristic X1)**

In the event of serious problems, the inverter default parameters can be recovered as follows:

Press PRG/RESET key, **0.Fnc** should be displayed.

Press ∧ key until **1.H__** is displayed.

Press FUNC/DATA key.

Press ∧ key until **H 03** is displayed.

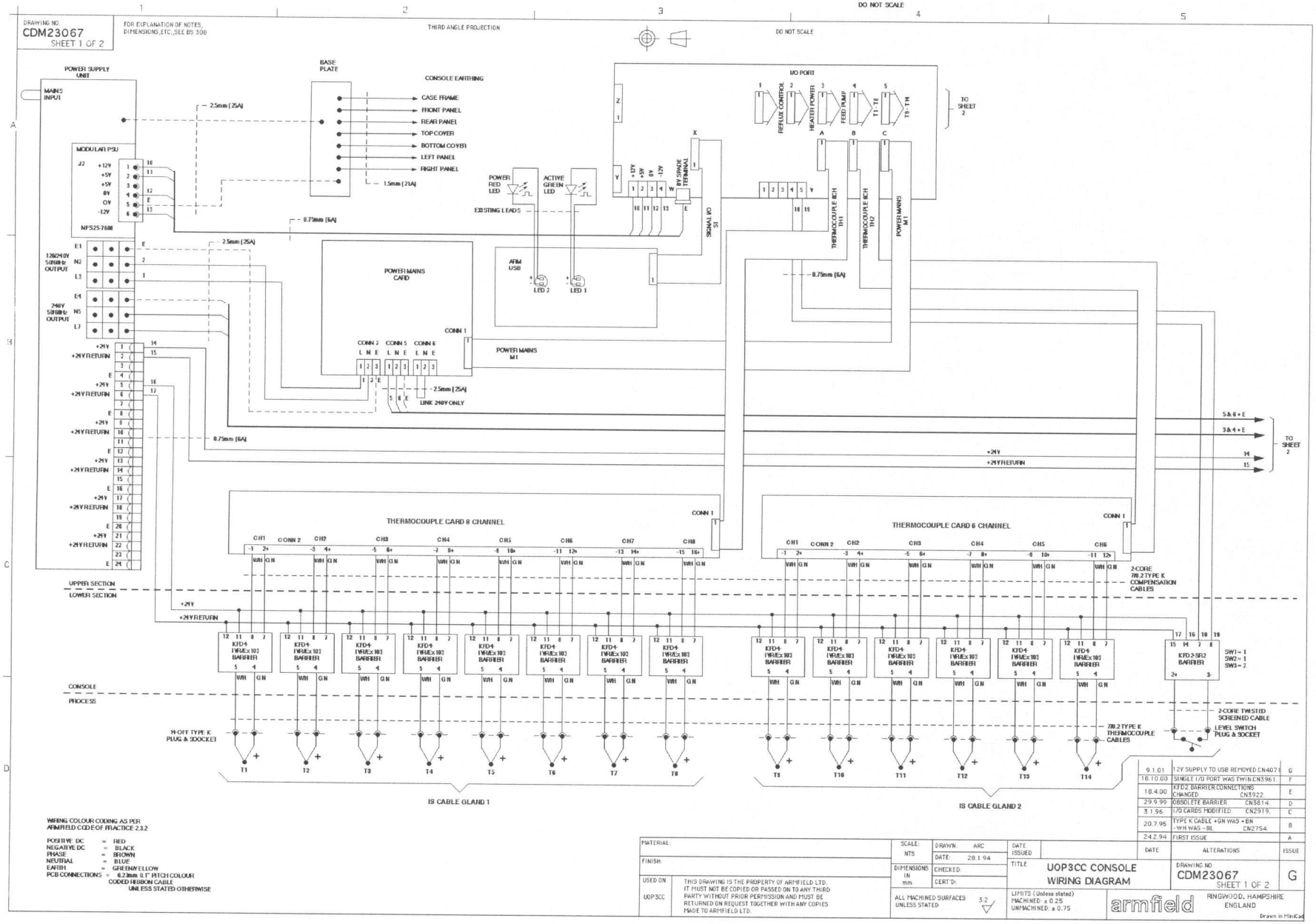
Press ∧ and **STOP** keys together.

Ensure that the display shows data value **1** (Initialize factory defaults).

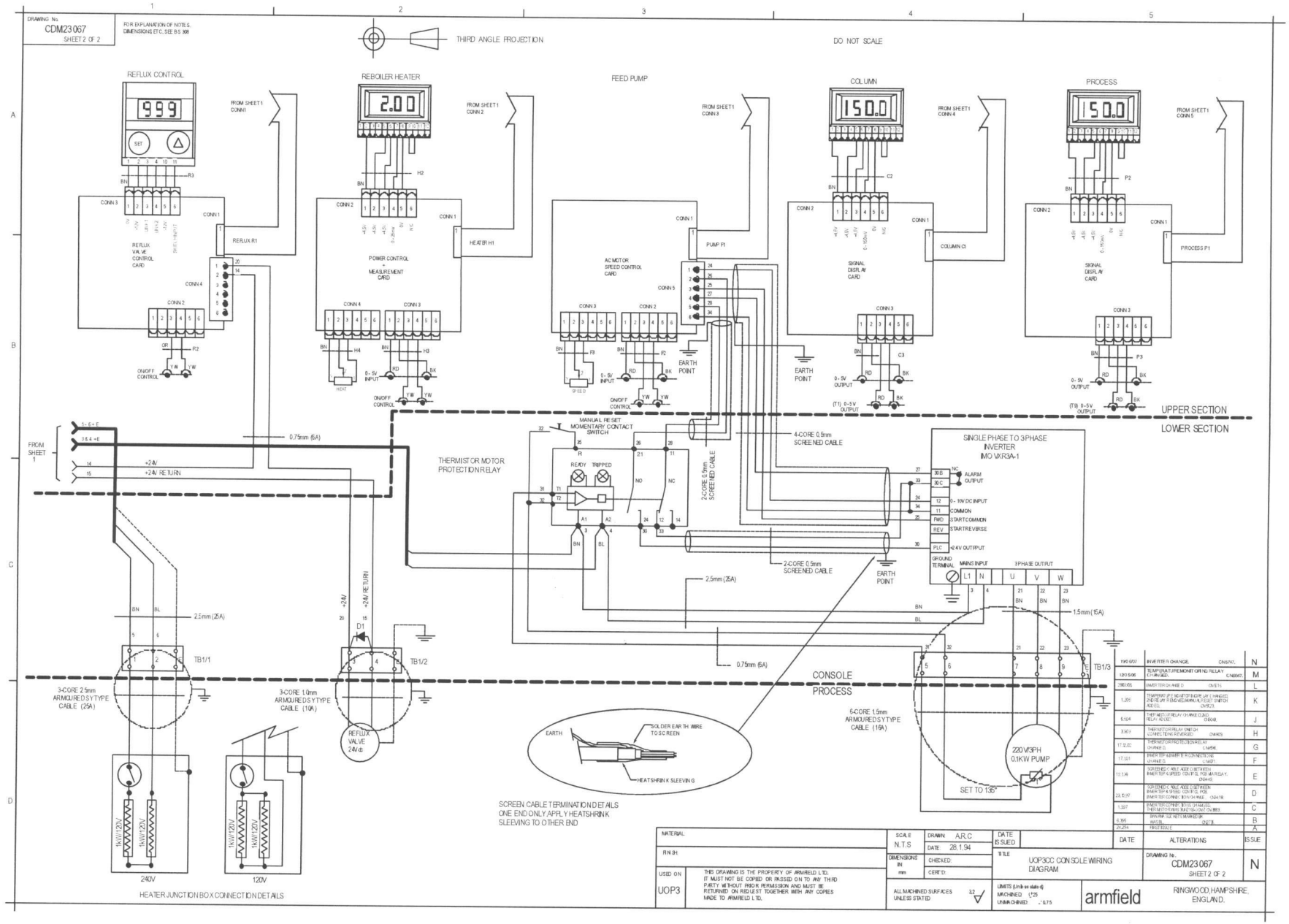
Press FUNC/DATA key.

Press PRG/RESET to return to normal operation

Refer to the inverter instruction manual supplied with UOP3CC (IMO Jaguar VXR3A-1) for a detailed explanation of all inverter parameters and parameter setting methods.


DRAWING NO.
CDM23067
SHEET 1 OF 2
FOR EXPLANATION OF NOTES, DIMENSIONS, ETC., SEE BS 308
THIRD ANGLE PROJECTION
DO NOT SCALE
POWER SUPPLY UNIT
MAINS INPUT
MODULAR PSU
NFS25-7608
BASE PLATE
CONSOLE EARTHING
CASE FRAME
FRONT PANEL
REAR PANEL
TOP COVER
BOTTOM COVER
LEFT PANEL
RIGHT PANEL
2.5mm (2SA)
1.5mm (2IA)
0.75mm (6A)
I/O PORT
REFLUX CONTROL
HEATER POWER
FEED PUMP
T1 - T8
T9 - T14
TO SHEET 2
POWER RED LED
ACTIVE GREEN LED
EXISTING LEADS
BY SPADE TERMINAL
SIGNAL I/O SI
THERMOCOUPLE INCH TH1
THERMOCOUPLE INCH TH2
POWER MAINS M1
AFM USB
LED 2
LED 1
POWER MAINS CARD
CONN 1
CONN 2 L N E
CONN 5 L N E
CONN 6 L N E
LINK 240V ONLY
POWER MAINS M1
120/240V 50/60Hz OUTPUT
240V 50/60Hz OUTPUT
+24V
+24V RETURN
THERMOCOUPLE CARD 8 CHANNEL
CH1 CH2 CH3 CH4 CH5 CH6 CH7 CH8
WH GN
2-CORE 70/0.2 TYPE K COMPENSATION CABLES
UPPER SECTION
LOWER SECTION
KFD4 IMWEx103 BARRIER
KFD2-SR2 BARRIER
SW1 = 1
SW2 = 1
SW3 = 2
CONSOLE
PROCESS
14-OFF TYPE K PLUG & SOCKET
70/0.2 TYPE K THERMOCOUPLE CABLES
2-CORE TWISTED SCREENED CABLE
LEVEL SWITCH PLUG & SOCKET
T1 T2 T3 T4 T5 T6 T7 T8 T9 T10 T11 T12 T13 T14
IS CABLE GLAND 1
IS CABLE GLAND 2
WIRING COLOUR CODING AS PER ARMFIELD CODE OF PRACTICE 2.3.2
POSITIVE DC = RED
NEGATIVE DC = BLACK
PHASE = BROWN
NEUTRAL = BLUE
EARTH = GREEN/YELLOW
PCB CONNECTIONS = 0.23mm 0.1" PITCH COLOUR CODED RIBBON CABLE UNLESS STATED OTHERWISE
9.1.01 12V SUPPLY TO USB REMOVED CN4071 G
16.10.00 SINGLE I/O PORT WAS TWIN CN3961 F
18.4.00 KFD2 BARRIER CONNECTIONS CHANGED CN3922 E
29.9.99 OBSOLETE BARRIER CN3614 D
3.1.96 I/O CARDS MODIFIED CN2919 C
20.7.95 TYPE K CABLE +GN WAS +BN -WH WAS -BL CN2754 B
2.4.2.94 FIRST ISSUE A
DATE ALTERATIONS ISSUE
MATERIAL:
FINISH:
USED ON UOP3CC
THIS DRAWING IS THE PROPERTY OF ARMFIELD LTD. IT MUST NOT BE COPIED OR PASSED ON TO ANY THIRD PARTY WITHOUT PRIOR PERMISSION AND MUST BE RETURNED ON REQUEST TOGETHER WITH ANY COPIES MADE TO ARMFIELD LTD.
SCALE: NTS
DRAWN: ARC
DATE: 28.1.94
CHECKED:
CERT'D:
DIMENSIONS IN mm
ALL MACHINED SURFACES UNLESS STATED 3.2
DATE ISSUED
TITLE UOP3CC CONSOLE WIRING DIAGRAM
DRAWING NO CDM23067 SHEET 1 OF 2 G
LIMITS (Unless stated) MACHINED: ± 0.25 UNMACHINED: ± 0.75
armfield
RINGWOOD, HAMPSHIRE ENGLAND
Drawn in MiniCad

- Back of sheet 1 -


DRAWING No. CDM23 067 SHEET 2 OF 2
FOR EXPLANATION OF NOTES, DIMENSIONS ETC., SEE BS 308
THIRD ANGLE PROJECTION
DO NOT SCALE
REFLUX CONTROL
REBOILER HEATER
FEED PUMP
COLUMN
PROCESS
FROM SHEET 1 CONN1
FROM SHEET 1 CONN2
FROM SHEET 1 CONN3
FROM SHEET 1 CONN4
FROM SHEET 1 CONN5
REFLUX VALVE CONTROL CARD
POWER CONTROL + MEASUREMENT CARD
AC MOTOR SPEED CONTROL CARD
SIGNAL DISPLAY CARD
REFLUX R1
HEATER H1
PUMP P1
COLUMN C1
PROCESS P1
ON/OFF CONTROL
0 - 5V INPUT
0 - 5V OUTPUT
EARTH POINT
UPPER SECTION
LOWER SECTION
FROM SHEET 1
+24V
+24V RETURN
0.75mm (6A)
THERMISTOR MOTOR PROTECTION RELAY
MANUAL RESET MOMENTARY CONTACT SWITCH
READY
TRIPPED
4-CORE 0.5mm SCREENED CABLE
2-CORE 0.5mm SCREENED CABLE
SINGLE PHASE TO 3 PHASE INVERTER IMO VXR3A-1
ALARM OUTPUT
0 - 10V DC INPUT
COMMON
START COMMON
START REVERSE
+24 V OUTPUT
GROUND TERMINAL
MAINS INPUT
3 PHASE OUTPUT
L1
N
U
V
W
2.5mm (25A)
1.5mm (15A)
TB1/1
TB1/2
TB1/3
3-CORE 2.5mm ARMOURED SWA TYPE CABLE (25A)
3-CORE 1.0mm ARMOURED SWA TYPE CABLE (10A)
6-CORE 1.5mm ARMOURED SWA TYPE CABLE (16A)
REFLUX VALVE 24V dc
CONSOLE PROCESS
220 V/3PH 0.1KW PUMP
SET TO 135°
EARTH
SOLDER EARTH WIRE TO SCREEN
HEAT SHRINK SLEEVING
SCREEN CABLE TERMINATION DETAILS ONE END ONLY APPLY HEATSHRINK SLEEVING TO OTHER END
1KW/120V
240V
120V
HEATER JUNCTION BOX CONNECTION DETAILS
MATERIAL
FINISH
USED ON
UOP3
THIS DRAWING IS THE PROPERTY OF ARMFIELD LTD. IT MUST NOT BE COPIED OR PASSED ON TO ANY THIRD PARTY WITHOUT PRIOR PERMISSION AND MUST BE RETURNED ON REQUEST TOGETHER WITH ANY COPIES MADE TO ARMFIELD LTD.
SCALE N.T.S
DRAWN A.R.C
DATE 28.1.94
CHECKED
CERT'D
DIMENSIONS IN mm
ALL MACHINED SURFACES UNLESS STATED 3.2
DATE ISSUED
TITLE UOP3CC CONSOLE WIRING DIAGRAM
DRAWING No. CDM23 067 SHEET 2 OF 2
LIMITS (Unless stated) MACHINED ±.25 UNMACHINED ±.0.75
armfield
RINGWOOD, HAMPSHIRE, ENGLAND.
DATE
ALTERATIONS
ISSUE
INVERTER CHANGE
TEMPERATURE MONITORING RELAY CHANGED
THERMISTOR CHANGED
TRANSFORMER, MOMENTARY CONTACT SWITCH, REFLUX RELAY, MANUAL RESET SWITCH ADDED
THERMISTOR RELAY CHANGED
THERMISTOR RELAY SWITCH CONNECTIONS REVISED
THERMISTOR PROTECTION RELAY CHANGED
INVERTER & MOTOR CONNECTIONS CHANGED
SCREENED CABLE ADDED BETWEEN INVERTER & SPEED CONTROL POT, INVERTER & SPEED CONTROL BOARD, PCB AND RELAY K
INVERTER CONNECTIONS CHANGED, THERMISTOR WIRE REACTIVATED
WIRING SIZE NOTE MARKED ON DRAWING
FIRST ISSUE

- Back of sheet 2 -

6 Troubleshooting

If the equipment does not function correctly it is suggested that the following checks are carried out.

6.1 If the equipment fails to operate at all, and the console displays are not illuminated:

6.1.1 RCD Check

Ensure that the Residual Current Device is latched.

If the RCD trips in operation or fails to latch then a fault is indicated inside the electrical console or in the electrical equipment installed on the distillation column. This fault condition could be dangerous to the operator and the equipment must not be operated until checked by a competent electrician.

6.1.2 MCB Check

Ensure that all three Miniature Circuit Breakers are latched. If one or more of the miniature circuit breakers trips in operation or fails to latch then a fault is indicated inside the electrical console or in the electrical equipment installed on the distillation column.

6.1.3 Electrical Supply Check

Ensure that the Liquid Crystal Displays associated with the instrumentation operate when the RCCB is latched (Red 'ON' LED on Temperatures should also be illuminated).

If displays and 'ON' LED are not illuminated check that an electrical supply of appropriate voltage (as described in the Specifications section of this manual) is connected to the console and the supply is switched on.

If the above check confirms that the console is connected to an appropriate electrical supply then a fault is likely in the dc Power Supply located inside the console. The dc Power Supply incorporates a fuse to protect the mains supply. This should be replaced with a fuse of appropriate rating as follows:

Fuse type Quickblow (F), 20mm x 5mm diameter, 2 Amps

6.2 If the console displays are illuminated but the console does not function correctly:

6.2.1 Selector switch setting check

If any of the console functions appears to malfunction firstly check that the selector switch is set to the appropriate position:

MANUAL The appropriate controlled variable is adjusted via the front panel control e.g. motor speed is varied by rotating the multi-turn potentiometer

INPUT SOCKET The appropriate controlled variable is adjusted by a 0-5 Vdc signal applied at the Red and Black sockets on the front panel e.g. PID control of reboiler power using PCT20H

ON/OFF The appropriate controlled variable is switched on or off by a switched input connected to the yellow sockets on the front panel.

When switched ON the appropriate variable can be adjusted via the front panel control e.g. On/off control of reboiler power using the relay output on PCT20H. Actual power can be varied by rotating the multi-turn potentiometer

I/O PORT The appropriate controlled variable is adjusted by a PC connected to the USB port at the rear of the console.

6.2.2 Console function check

REFLUX CONTROL Ensure that the Red 'ON' LED is illuminated. If not then press the momentary action switch to switch the function on. If the Red LED fails to illuminate then a fault exists in the Reflux Control PCB inside the console.

If the selector switch is set for manual operation then ensure that the timer has been configured correctly and appropriate time periods have been set for operation of the reflux valve, as described in the Operation section of this manual.

REBOILER HEATER Ensure that the Red 'ON' LED is illuminated. If not then press the momentary action switch to switch the function on. If the Red LED fails to illuminate then a fault exists in the Reboiler Heater PCB inside the console.

If the 'LOW LEVEL' LED is illuminated then a low level exists in the reboiler or the level switch is disconnected. For safe operation, the reboiler heater cannot be switched on until the heating element is covered.

If using the Red & Black Input sockets for connection to a PID controller ensure that the selector switch is set to INPUT SOCKET and the input signal is in the range 0-5Vdc

If using the yellow Input On/off sockets for connection to an on/off controller ensure that the selector switch is set to ON/OFF and the multi-turn potentiometer has been set to the required reboiler power.

FEED PUMP

Ensure that the green 'ON' LED is illuminated. If not then press the momentary action switch to switch the function on.

If the 'TRIP' LED illuminates this indicates a fault in the speed controller or overheating of the motor associated with the feed pump. Check the pump for obvious signs of a fault, such as overheating, seized rotor, damaged tubing etc. To reset the speed controller, briefly disconnect the supply to the console by pressing the test button on the RCCB then latching the RCCB on.

If the 'TRIP' LED continues to illuminate then allow the motor to cool before resetting. A failure to reset should be checked by a competent electrician.

If using the Red & Black Input sockets for connection to a PID controller ensure that the selector switch is set to INPUT SOCKET and the input signal is in the range 0-5Vdc.

If using the yellow Input On/off sockets for connection to an on/off controller ensure that the selector switch is set to ON/OFF and the multi-turn potentiometer has been set to the required pump speed.

COLUMN TEMPERATURES

If the Red 'ON' LED fails to illuminate then a fault exists in the Temperature PCB inside the console.

Ensure that the top selector switch has been set to indicate the required temperature on the Liquid crystal display.

If any temperature reading is not sensible, firstly check that the tip of the thermocouple is immersed in the liquid on the sieve plate, then check that the correct thermocouple is connected to the miniature thermocouple connector on the Distillation Column. A faulty thermocouple can be checked by substituting an alternative thermocouple - if the reading is restored then the original thermocouple is faulty. If the error continues then the calibration of the thermocouple should be checked. Each thermocouple has corresponding zero and span potentiometers accessible through holes in the rear panel of the electrical console.

If using the upper red and black output sockets for connection to a chart recorder, data logger or controller then the lower selector switch should be set to output the required temperature. 5Vdc = 150 degrees Celsius.

If using the lower red and black output sockets for connection to a chart recorder, data logger or controller then temperature T1 is permanently output. 5Vdc = 150 degrees Celsius.

PROCESS TEMPERATURES If the green 'ON' LED fails to illuminate then a fault exists in the Process Temperature PCB inside the console.

Ensure that the top selector switch has been set to indicate the required temperature on the Liquid crystal display.

If any temperature reading is not sensible check that the correct thermocouple is connected to the miniature thermocouple connector on the distillation column.

A faulty thermocouple can be checked by substituting an alternative thermocouple - if the reading is restored then the original thermocouple is faulty. If the error continues then the calibration of the thermocouple should be checked. Each thermocouple has corresponding zero and span potentiometers accessible through holes in the rear panel of the electrical console.

If using the upper red and black output sockets for connection to a chart recorder, data logger or controller then the lower selector switch should be set to output the required temperature. 5Vdc = 150 degrees Celsius

If using the lower red and black output sockets for connection to a chart recorder, data logger or controller then temperature T9 is permanently output. 5Vdc = 150 degrees Celsius

6.3 If the console appears operational but the process does not behave as expected:

6.3.1 Valve position check

Ensure that all valves on the Distillation Column are set in the required positions to suit the demonstration in progress as stated in the appropriate sections of this manual.

If vapour escapes from the vent pipe, ensure that the cooling water is turned on (open valve V5) and check that the power to the reboiler is not too high. Switch off the reboiler power if any doubt exists.

The valve at the base of the decanter (V10) should be open at all times for normal operation.

6.4 Further assistance:

Any required minor electrical work should be carried out by a competent electrician. Wiring diagrams are supplied in this manual to assist in tracing and solving electrical problems.

Any other problems should be referred to Armfield. Contact your local Armfield agent. Alternatively, contact details are provided at the back of this manual.

7 Laboratory Teaching Exercises

7.1 Index to Exercises

7.2 Glossary of Terms

Flammable material. A gas, vapour, liquid, dust or solid that can react continuously with atmospheric oxygen and that may therefore sustain fire or explosion when such reaction is initiated by a suitable spark, flame or hot surface.

Explosive gas-air mixture. A mixture of flammable gas or vapour with air under atmospheric conditions in which, after ignition, combustion spreads throughout the unconsumed mixture.

Hazard. The presence, or the risk of the presence, of an explosive gas-air mixture.

Hazardous area. An area in which explosive gas-air mixtures are, or may be expected to be, present in quantities such as to require special precautions for the construction and use of electrical apparatus.

Non-hazardous area. An area in which explosive gas-air mixtures are not expected to be present so that special precautions for the construction and use of electrical apparatus are not required.

Source of release. A point from which a flammable gas, vapour or liquid may be released into the atmosphere.

Flash point. The lowest temperature at which sufficient vapour is given off from a flammable material to form an explosive gas-air mixture.

Ignition temperature. The lowest temperature of a flammable gas or vapour at which ignition occurs under test conditions specified in IEC Publications 79 - 4 and BS 4056.

Maximum surface temperature. The highest temperature attained under practical conditions of operation within the rating of the apparatus by an accessible surface the exposure of which to an explosive atmosphere may involve a risk.

Temperature class (T class). A system of classification by which an electrical apparatus is allocated one of six temperature classes according to its maximum surface temperature.

Grouping of apparatus. For some types of protection it is necessary for practical reasons to group apparatus according to detailed design characteristics.

Electrical apparatus complying with the requirements of a particular group may be used with all flammable gases and vapours associated with that group, or with materials of lesser risk, subject only to considerations of temperature classification and chemical compatibility.

Chemical Compatibility. The materials used for the construction of the apparatus and its installation should be chosen having regard to the solvent and corrosive agencies that may be present.

Intrinsic Safety. Intrinsic Safety is a technique for ensuring that the electrical energy available in a circuit is too low to ignite the most easily ignitable mixture of gas and air.

Zener Safety Barrier. A device, mounted in the non-hazardous area, to stop excess electrical energy from entering the hazardous area. (These devices are installed inside the electrical console).

7.3 Nomenclature

Name	Symbol	Units	Calculation
Temperature at top of column	T1	°C	
Temperature in second tray	T2	°C	
Temperature in third tray	T3	°C	
Temperature in fourth tray	T4	°C	
Temperature in fifth tray	T5	°C	
Temperature in sixth tray	T6	°C	
Temperature in seventh tray	T7	°C	
Temperature at bottom of column	T8	°C	
Temperature in reboiler	T9	°C	
Temperature of column exit vapour	T10	°C	
Cooling water entry temperature	T11	°C	
Cooling water exit temperature	T12	°C	
Condensate/top product temperature	T13	°C	
Cooling water flow rate	FI1	l/hr	
Total pressure drop across column	ΔP1	mm H_2O	
Vacuum pressure	P1	BAR	
Vacuum pump pressure relief valve	PRV1	-	
Boiler heater power		kW	
Boil-up rate		l/hr	$\frac{\text{Condensate volume collected}}{\text{Time to collect}}$
Continuous feed valve	V1	-	
Reboiler sample extraction valve	V2	-	
Condenser sample extraction valve	V3	-	
Top product receiver sample valve	V4	-	
Cooling water flow control valve	V5	-	
Bottom manometer connection	V6	-	
Top manometer connection	V7	-	
Dosing vessel floe control valve	V8	-	
Condensate decanter bypass valve	V10	-	
Drain valve	V11	-	

Product recycling valve	V12	-
Vacuum pump water supply control	V14	-
Vacuum pump flow control valve	V15	-

7.4 Exercise A: Variation of Column Pressure Drop

Objective

To determine the variation with boil-up rate of pressure drop over the distillation column.

Method

By setting the equipment to operate at total reflux (meaning all the formed vapour returns to the column after condensation); the pressure drop over the distillation column is then measured for a variety of different boil-up rates.

Equipment Required

Armfield UOP3CC

5.43 litres Methylcyclohexane

4.57 litres Toluene

250ml measuring cylinder graduated in ml

Stopwatch (or similar timing device)

Theory

The total pressure drop across each tray is the sum of that caused by the restriction of the holes in the sieve tray, and that caused by passing through the liquid (foam) on top of the tray.

As the velocity of the vapours passing up the column increases then so does the overall pressure drop. The velocity is controlled by varying the boil-up rate which is done by varying the power input to the reboiler. Under conditions with no liquid present, the sieve trays will behave like an orifice in that the pressure drop will be proportional to the square of the velocity. Due to the fact that there is a liquid head however, this square relationship does not become apparent until the head of liquid has been overcome and foaming is taking place. In a graph of pressure drop vs. boil-up rate (log/log), at low boil-up rates the pressure drop will remain fairly constant until foaming occurs when the pressure drop would be expected to rise sharply for unit increases in boil-up rate.

Equipment Set Up

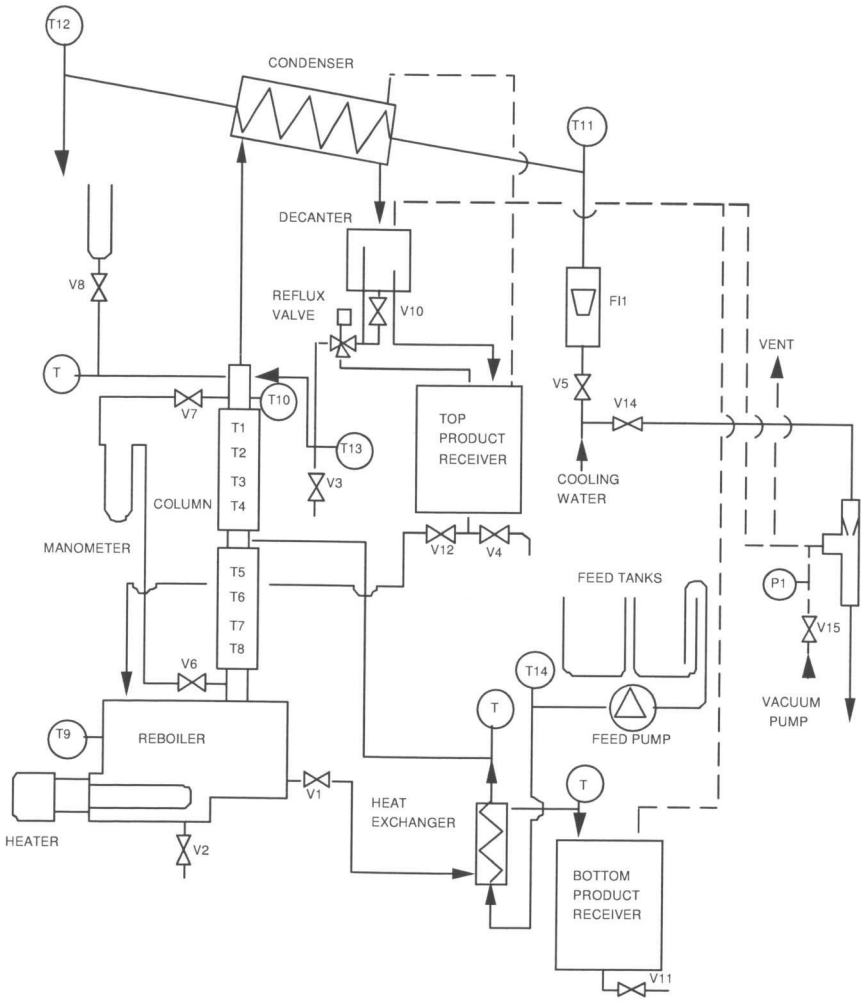
Make up 10 litres of a mixture of 50 mol percent methyl*cyclo*hexane and 50 mol percent toluene. Required for this mixture are:

$$\frac{50 \times 98.19}{0.774} : \frac{50 \times 92.15}{0.867}$$

6343.0:5314.3

1.19:1

Thus for 10 litres there are 5.43 litres of methyl*cyclo*hexane and 4.57 litres of toluene.



Set the equipment to operate at total reflux by switching off the reflux ratio timer on the console.

Ensure all valves on the equipment are closed.

Open valve V10 on the reflux pipe.

Load the 10 litre charge of prepared feed mixture (the liquid to be distilled) directly into the reboiler through the filler cap provided. Make sure the filler cap on the top of the reboiler is firmly replaced.

Procedure

Turn on the power to the control panel.

Set the temperature selector switch to T9 (the temperature in the reboiler).

Open valve V5 until the cooling water flow rate FI1 to the condenser is approximately 3 litres/min.

On the control panel, turn the power controller for the reboiler heating element on full (fully anti-clockwise) and switch the switch turning on the power to the heating element to "power on" position. Another red lamp will illuminate indicating the heating element is on.

Turn the power controller clockwise until a reading of approximately 0.75kW is obtained on the digital wattmeter. The contents of the reboiler will begin to warm up and this can be observed on the temperature readout meter.

Open valves V6 and V7 which connect base and top of the distillation column, respectively, to the manometer. Initially there will be no pressure difference in the column meaning no reading on the manometer. Close valve V6 and V7.

The 1st and 5th plates in the column sections are not insulated so that observation of the sieve plates is possible. Make notes on the appearance of these trays throughout the distillation process.

Eventually, vapour will begin to rise up the column and the progress of this can be clearly observed as well as detected by the increasing temperatures when switching the temperature selector on T8, T7, T6, T5, T4, T3, T2 and T1. Vapour will enter the condenser and reappear as droplets into the glass walled distillate receiver vessel (11). The distillate will build up a small level in the receiver and eventually overflow to the reflux regulator valve (12). Since the valve is not on (on the control panel), and it will not be necessary to have it on in this experiment, which is run under total reflux, the condensed vapour will return to the column.

The cool distillate is then returning on the top of the column, and will cascade down the trays forming a liquid level on the trays and bubbling of vapour passing through the liquid. The system will have reached an equilibrium condition when the temperatures T1, T2, T3, T4, T5, T6, T7 and T8 are constant.

Measure the boil-up rate by performing a timed volume collection: Operate valve V3 so that all the condensate is diverted into a measuring cylinder, and use the stopwatch to measure the time required to collect a set quantity. This will not disrupt the equilibrium conditions in the column provided a liquid level is maintained in the condensate feeding pipe. When taking a sample, partially open valve V3 and drain the condensate (in a separate measuring cylinder) from the reflux system until a steady flow is obtained. (Ensure that liquid remains in the flexible connecting tube to prevent vapour from escaping.) Start sample collection and timing at the same time, and collect a sizeable amount. Pour the first non-representative collected amount in a bottle labelled "recyclable MHC/toluene".

After taking the samples, take readings of pressure drops over both the rectifying (top) and the stripping (bottom) sections by opening the valves V6 and V7 on the manometer. When opening the valves, make sure always to open valve V6 then V7 to

prevent vapour from the column entering the manometer. If this happens, it can be seen as two separate phases in the U-tube. Close the valves in the same order after taking the pressure drop reading.

Repeat these readings until two in a row agree fairly closely. Allow 5 to 10 minutes between each set of measurements before starting the next set in order for the system to reach equilibrium again.

Step up the boil-up rate in 250 Watt increments up to maximum 1.5 kW by adjusting the boiler heater power controller (on the control panel). Take similar readings of the boil-up rate and pressure drops, after having allowed at least 10 minutes to allow the column to stabilise.

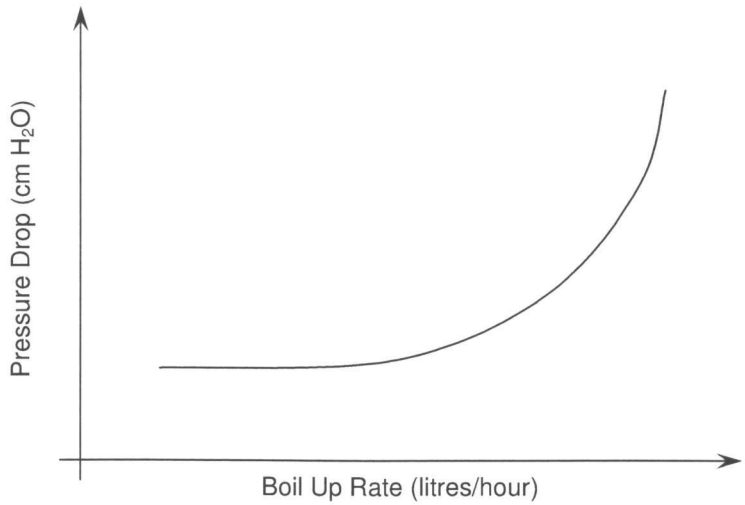
Results

Tabulate your results under the following headings:

Power (kW)	Boil-up Rate (litres/hr)	Pressure Drop (cm H2O) Top Bottom Overall	Degree of Foaming On Trays
0.50			
0.75			
1.00			
1.25			
1.50			
1.75			

The column headed 'Degree of Foaming on Trays' should be filled in using descriptive words, e.g. 'none', 'gentle localised', 'violent localised', 'foaming gently over whole tray', 'foaming violently over whole tray', 'liquid flooding in column'.

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



Conclusion

Comment on the relationship observed, if any, between boil-up rate and pressure drop.

Relate the descriptive comments observed to zones on the curve.

7.5 Exercise B: Determining Mixture Compositions

Objective

To use a refractometer to determine mixture compositions.

Method

A selection of difference mol percentage binary mixtures are made up and the refractive index of each is measured.

Equipment Required

UOP3CC

Hand held refractometer

0 – 100 ml measuring cylinder

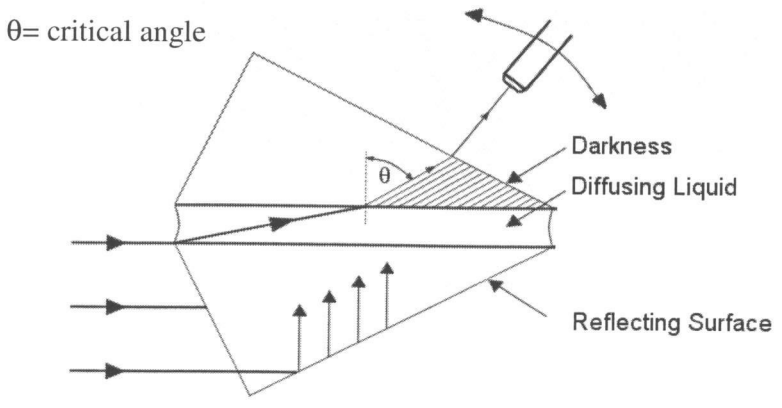
A suitable container to mix the samples in

A bottle of methylcyclohexane

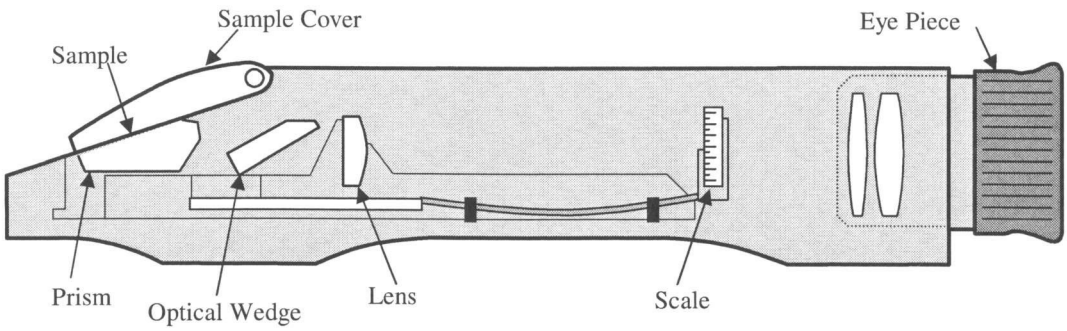
A bottle of toluene

Theory

For the system methyl*cyclo*hexane/toluene, mixtures of known concentration can be made up and their refractive indexes measured. The refractometer measures the critical angle of the liquid under test and each concentration will show a different critical angle.



Beyond the critical angle is darkness, and refractometers are calibrated along this light/dark boundary.



Hand held refractometer

Equipment Set Up

Make up small quantities of 25 mol percent, 50 mol percent and 75 mol percent methyl*cyclo*hexane. Calculate the volume of constituents to use as follows:

For 25 mol percent methyl*cyclo*hexane i.e. 75 mol percent toluene

molecular weight methyl*cyclo*hexane = 98.19

molecular weight toluene = 92.15

density methyl*cyclo*hexane = 0.774 g/ml

density toluene = 0.867 g/ml

$$25 = \frac{\frac{\text{Vol}_{\text{MCH}} x \rho_{\text{MCH}} x 100}{\text{MW}_{\text{MCH}}}}{\frac{\text{VOL}_{\text{MCH}} x \rho_{\text{MCH}}}{\text{MW}_{\text{MCH}}} + \frac{\text{Vol}_{\text{tol}} x \rho_{\text{tol}}}{\text{MW}_{\text{tol}}}}$$

thus

$$25 = \frac{100}{1 + \frac{\text{Vol}_{\text{tol}}}{\text{Vol}_{\text{MCH}}} X \frac{\text{MW}_{\text{MCH}}}{\text{MW}_{\text{tol}}} x \frac{\rho_{\text{tol}}}{\rho_{\text{MCH}}}}$$

$$\frac{\text{Vol}_{\text{tol}}}{\text{Vol}_{\text{MCH}}} x \frac{\text{MW}_{\text{MCH}}}{\text{MW}_{\text{tol}}} x \frac{\rho_{\text{tol}}}{\rho_{\text{MCH}}} = 3$$

$$\frac{\text{Vol}_{\text{tol}}}{\text{Vol}_{\text{MCH}}} = 3X \frac{\text{MW}_{\text{tol}}}{\text{MW}_{\text{MCH}}} x \frac{\rho_{\text{MCH}}}{\rho_{\text{tol}}}$$

$$\frac{\text{Vol}_{\text{tol}}}{\text{Vol}_{\text{MCH}}} = 3X \frac{92.15}{98.19} X \frac{0.774}{0.867}$$

$$1\ \text{Vol}_{\text{tol}} = 2.51\ \text{Vol}_{\text{MCH}}$$

Thus for 100 mls of mixture, quantities required will be

28.49 ml methyl*cyclo*hexane

71.51 ml toluene

Procedure

Using the refractometer, measure the refractive index (R.L.) of pure methyl*cyclo*hexane and pure toluene (follow the manufacturer's instructions for the refractometer to be used).

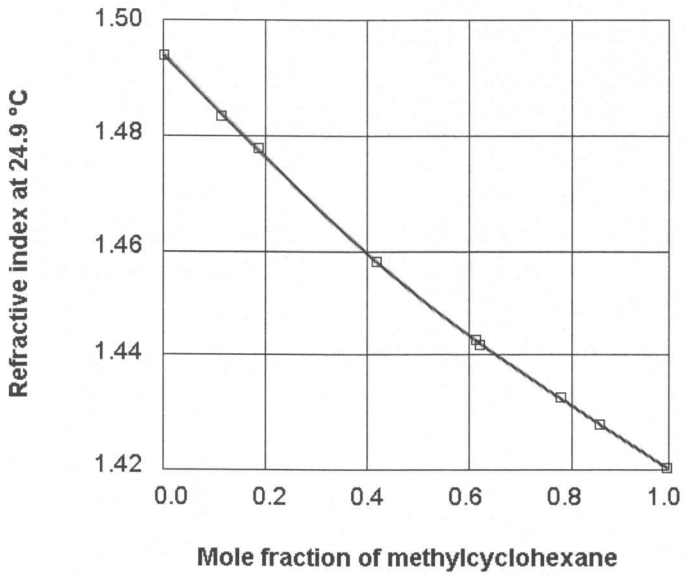
Measure the R.I. of the 25%, 50% and 75% mol percent methylcyclohexane/toluene mixtures.

Results

Tabulate your measurements under the following headings:

Methyl*cyclo*hexane Concentration	**R.I.**
100%	
75%	
50%	
25%	
0%	

Plot a graph of refractive index versus mol percent methyl*cyclo*hexane in the methyl*cyclo*hexane/toluene mixture. The results should be similar to the following example.



Conclusion

The results obtained from this experiment allow the mixture composition to be determined from a measurement of the refractive index. For any mixture composition of these two constituents, measure the refractive index (plotted on the y-axis) and read off the composition (from the x-axis). Comment on the probable accuracy of the graph obtained, and suggest how accuracy could be improved.

7.6 Exercise C: Overall Column Efficiency

Objective

To determine the overall column efficiency.

Method

To operate the column over a range of boil-up rates, taking samples across the range in order to measure the refractive index (and hence to determine the concentration of the components).

Equipment Required

UOP3CC

4.57 litres of toluene

5.43 litres of methyl*cyclo*hexane

250 ml measuring cylinder graduated in ml

Stop watch or similar timing device

Hand held refractometer (not supplied)

Refractive index/concentration calibration graph from Exercise B

Theory

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

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

where

n	=	number of theoretical plates
x_A	=	mole fraction of more volatile component
x_B	=	mole fraction of least volatile component
a_{av}	=	average relative volatility

Subscripts D, B indicate distillate and bottom respectively

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

The efficiency is given by

$$E = \frac{\text{Number of theoretical plates}}{\text{Number of actual plates}} x100\%$$

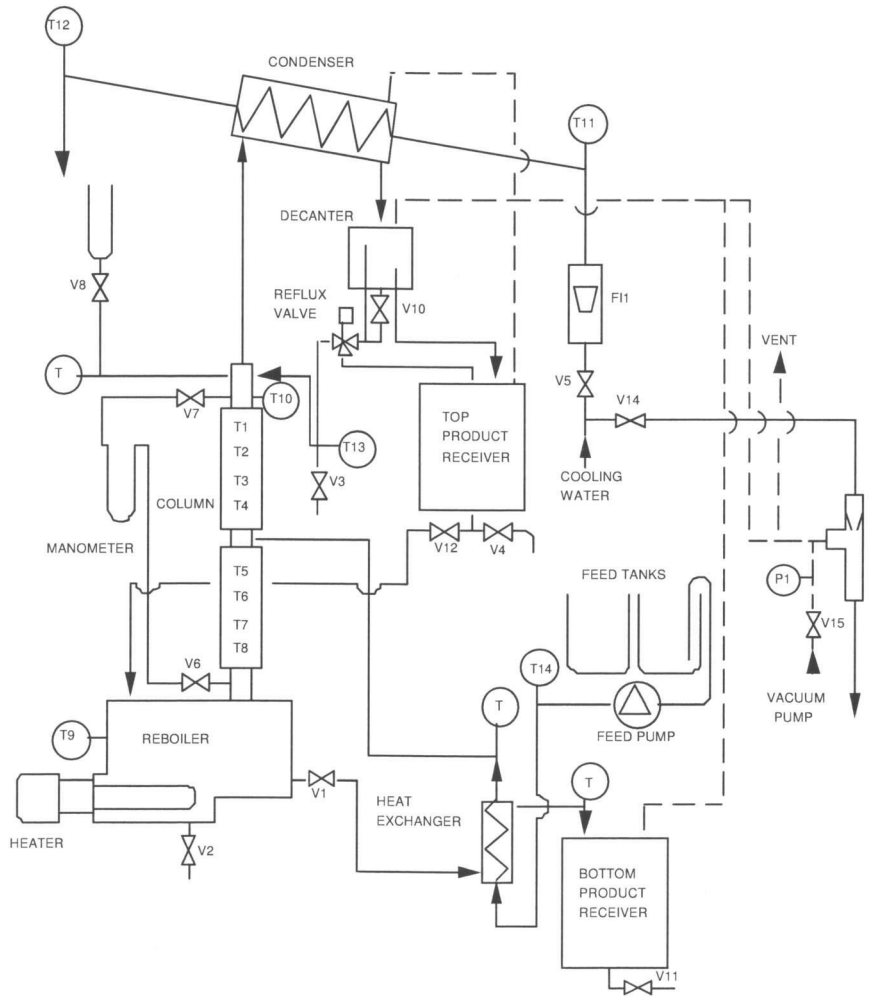
Knowing the composition of distillate and bottom and the corresponding volatilities, the column efficiency can be determined.

Equipment Set Up

Make up 10 litres of a mixture of 50 mol percent methyl*cyclo*hexane and 50 mol percent toluene. Required for the mixture are:

4.57 litres of toluene

5.43 litres of methyl*cyclo*hexane



Set the equipment to operate at total reflux by switching off the reflux ratio timer on the console.

Load the 10 litre charge of prepared feed mixture (the liquid to be distilled) directly into the reboiler through the filler cap provided. Make sure the filler cap on the top of

the reboiler is firmly replaced. At total reflux there will be no feed or top product or bottom product.

Procedure

Set the heat controller high then reduce heat as reflux is introduced to give steady bubbling on all tray and total reflux.

Leave the apparatus for at least 30 minutes so that the system can reach an equilibrium condition.

Measure the boil-up rate by performing a timed volume collection: Operate valve V3 so that all the condensate is diverted into a measuring cylinder. First take a small discarding sample of 5 to 10 ml into an alternative vessel, then divert the condensate into the measuring cylinder and use the stopwatch to measure the time required to collect a set quantity. This will not disrupt the equilibrium conditions in the column provided a liquid level is maintained in the condensate feeding pipe. When taking a sample, partially open valve V3 and drain the condensate (in a separate measuring cylinder) from the reflux system until a steady flow is obtained. (Ensure that liquid remains in the flexible connecting tube to prevent vapour from escaping.) Start sample collection and timing at the same time, and collect a sizeable amount. Repeat the measurement three times and take an average value to determine the boil-up rate.

After the representative sample has been taken, keep the sample glasses in an upright position. Do not overturn them because of the possibility of evaporation of the sample. Dispose of the discarding samples in a safe manner.

Measure and record the refractive index for the overhead samples taken.

Following a similar procedure, take a sample of the bottom through valve V2.

CAUTION! THIS SAMPLE WILL BE HOT!

Record the refractive index for this sample, too.

Repeat this procedure every ten minutes until five samples of both overhead and bottom are obtained. Record the temperatures T8 and T1 to calculate the average column temperature.

Repeat this procedure for several different boil-up rates to cover over the operating range of the column.

The calibration graph developed in Experiment B can be used to determine the concentrations of the components in the taken samples.

Results

Boil-up rate = _____ litres/hour.

Overhead Composition $(X_A)_d$ = a)
(mol percent methyl*cyclo*hexane) b)
c)
d)
e)

Average = _____ mol percent methyl*cyclo*hexane

Bottom Composition $(X_A)_b$ = a)
b)
c)
d)
e)

Average top column temperature = _____ °C
Average bottom column temperature = _____ °C
Average column temperature = _____ °C

Sample Calculation

For the first sample $(X_A)_d = 0.75$, $(X_B)_d = 0.25$
$(X_A)_b = 0.43$, $(X_B)_b = 0.57$,
where A = methyl*cyclo*hexane, and
B = toluene.

Find corresponding values of y from equilibrium data then:-

$$\alpha_d = 1.168, \alpha_b = 1.3405$$

$$\alpha_{av} = \sqrt{(1.168x1.3405)}, \underline{\alpha_{av} = 1.25}$$

From Fenske's equation

$$n+1 = \frac{\log\left[\left(\frac{0.75}{0.25}\right) \quad \left(\frac{0.57}{0.43}\right)\right]}{\log 1.25}$$

$$n+1 = \frac{0.60}{0.097} = 6.2$$

$\therefore$ $\underline{n = 5.2}$

i.e. number of theoretical plates = 5.2

Similarly for each sample E = a)

b)

c)

d)

e)

$\therefore$ Average Efficiency = _____ %

Conclusion

Is there any obvious relationship between boil-up rate and column efficiency?

How does the efficiency of the column compare to full-scale columns in use in industry?

7.7 Exercise D: Distillation at Constant Reflux Ratio

Objective

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

Equipment Required

UOP3CC

4.57 litres of toluene

5.43 litres of methyl*cyclo*hexane

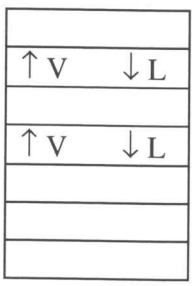
250 ml measuring cylinder graduated in ml

Stop watch (or similar timing device)

Hand held refractometer

Refractive index/concentration calibration graph from Exercise B

Theory



Since there is no feed, no bottom product or no top product, the liquid flow in the column is equal to the vapour flow in the column:

$$V = L$$

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

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

Since V = L this gives

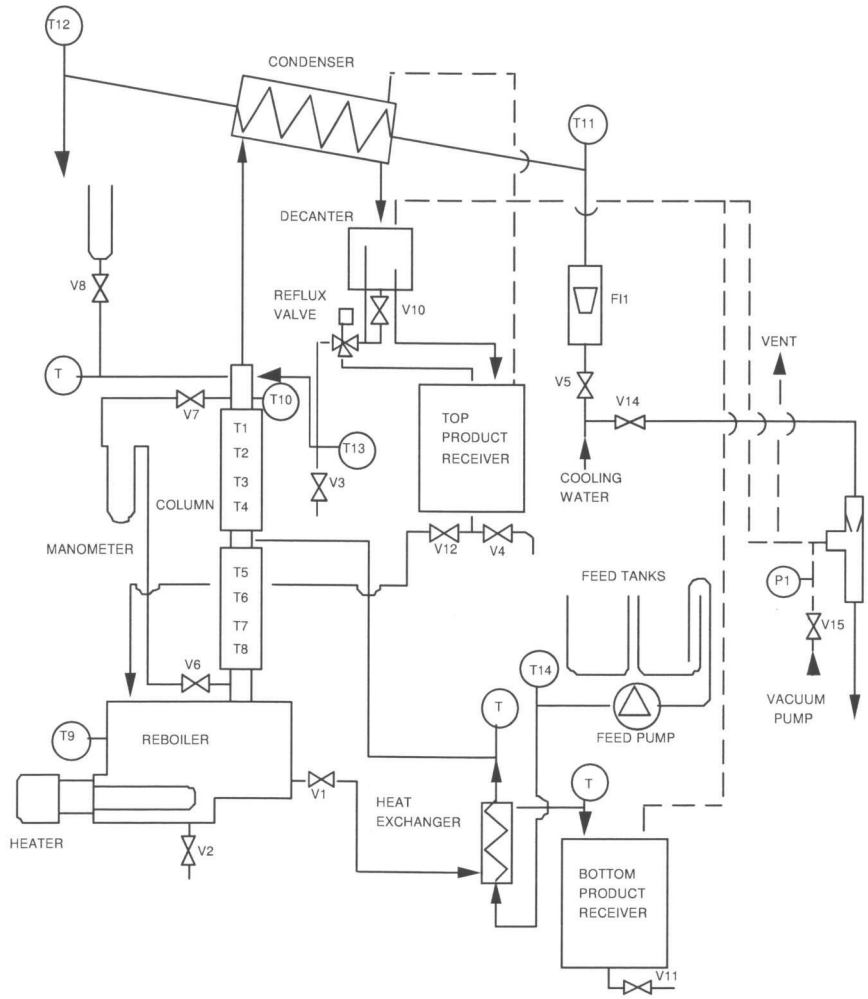
$$y_n = X_{n+1}$$

Equipment Set Up

Make up 10 litres of a mixture of 50 mol percent methyl*cyclo*hexane and 50 mol percent toluene. Required for the mixture are:

4.57 litres of toluene

5.43 litres of methyl*cyclo*hexane



Set the equipment to operate at total reflux by switching off the reflux ratio timer on the console.

Load the 10 litre charge of prepared feed mixture (the liquid to be distilled) directly into the reboiler through the filler cap provided. Make sure the filler cap on the top of the reboiler is firmly replaced. At total reflux there will be no feed or top product or bottom product.

Procedure

Set the heat controller high then reduce heat as reflux is introduced to give steady bubbling on all tray and total reflux. Leave the apparatus for at least 15 minutes so that the system can reach an equilibrium condition.

This experiment is run at varying reflux ratios. Set the reflux controller to the desired values e.g. start with 5:1 by setting 20 sec back to column and 4 sec to top product receiver. The setting is like the setting of a digital watch. C- means back to column and C+ means to top product receiver. When this is done, switch the reflux valve on (on the control panel). You should also see condensed vapour flowing to the top product receiver.

Measure the boil-up rate by performing a timed volume collection: Operate valve V3 so that all the condensate is diverted into a measuring cylinder. First take a small discarding sample of 5 to 10 ml into an alternative vessel, then divert the condensate into the measuring cylinder and use the stopwatch to measure the time required to collect a set quantity. This will not disrupt the equilibrium conditions in the column provided a liquid level is maintained in the condensate feeding pipe. When taking a sample, partially open valve V3 and drain the condensate (in a separate measuring cylinder) from the reflux system until a steady flow is obtained. (Ensure that liquid remains in the flexible connecting tube to prevent vapour from escaping.) Start sample collection and timing at the same time, and collect a sizeable amount. Repeat the measurement three times and take an average value to determine the boil-up rate.

Take a sample of the overheads through valve V3. When doing so be careful never to drain the condensate return line, i.e. partially open valve V3 to leave a small amount of liquid in the line all the time.

Generally, when taking samples, drain a "discarding" sample of approximately 5 to 10 ml before taking the representative sample in a sample glass. Do not drain too much of the "discarding" sample because of the disturbance of the mass balance. Discard the "discarding" sample in a safe way. After the representative sample has been taken, keep the sample glasses in an upright position. Do not overturn them because of the possibility of evaporation of the sample.

Record the refractive index for the taken overhead sample. In a similar manner **and preferably at the same time** take a sample of the bottom through valve V2.

CAUTION! THIS SAMPLE IS HOT!

Record the refractive index for this sample too.

Repeat this procedure every ten minutes until five samples of both overhead and bottom are obtained. Record the temperatures T8 and T1 to calculate the average column temperature.

Repeat this procedure for different reflux ratios.

Repeat the whole procedure for several different boil-up rates to cover over the operating range of the column.

The calibration graph developed in Experiment B can be used to determine the concentrations of the components in the taken samples.

Results

SAMPLE CALCULATION:

Top product required: 75 mol percent methyl*cyclo*hexane

Bottom product required: 37 mol percent methyl*cyclo*hexane

From the equilibrium curve,

Composition on the top sieve plate is yt = 0.78, xt = 0.75

and $y_{t-1} = x_t = 0.75$

$x_{t-1} = 0.72$

$y_{t-2} = x_{t-1} = 0.72$

$x_{t-2} = 0.69$

$y_{t-3} = x_{t-2} = 0.69$

$x_{t-3} = 0.65$

$y_{t-4} = x_{t-3} = 0.65$

$x_{t-4} = 0.60$

$y_{t-5} = x_{t-4} = 0.60$

$x_{t-5} = 0.54$

$y_{t-6} = x_{t-5} = 0.54$

$x_{t-6} = 0.47$

$y_{t-7} = x_{t-6} = 0.47$

$x_{t-7} = 0.40$

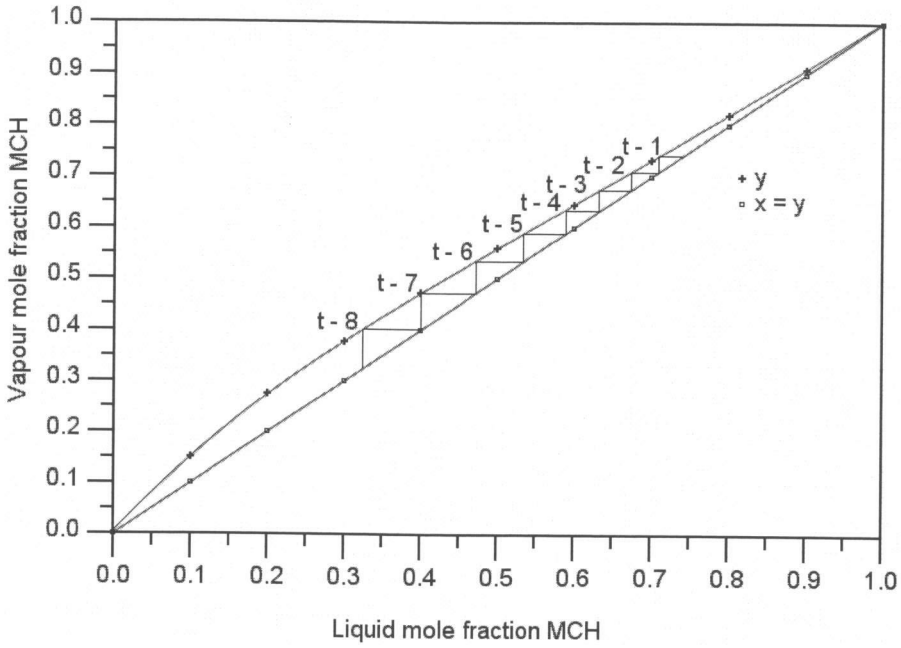
$y_{t-8} = x_{t-7} = 0.40$

$x_{t-8} = 0.37$

This composition is close to that required and can be withdrawn from the bottom product.

Theoretically, therefore, the distillation column containing eight sieve plates plus the boiler will give the compositions calculated above. However, as the experiment will show this is not in fact correct. Relate the theory and the results of the experiment with the previous experiment to determine column efficiency.

The previous calculation can be illustrated in a so-called McCabe-Thiele diagram:


Vapour mole fraction MCH
Liquid mole fraction MCH
1.0
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0.0
0.0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0.8
0.9
1.0
t - 1
t - 2
t - 3
t - 4
t - 5
t - 6
t - 7
t - 8
+ y
▫ x = y

Total condensate flow rate a)

b)

c)

d)

e)

Average flow rate =

Top Product Composition Mol Percent methylcyclohexane	Bottom Product Composition Mol Percent methylcyclohexane

Conclusion

Refer back to the Theory section of this experiment. Did the results obtained in this experiment correlate with the McCabe-Thiele diagram presented in the theory section?

7.8 Exercise E: Steady State Distillation of a Binary Mixture

Objective

To investigate the steady state distillation of a binary mixture under continuous operation.

Equipment Required

UOP3CC

65 mol % methyl*cyclo*hexane/ 35 mol % toluene

25 mol % methyl*cyclo*hexane/75 mol % toluene mixture

1 - 250 ml measuring cylinder graduated in mls.

1 - stop watch

1 - hand held refractometer

calibration curve for the feed pump

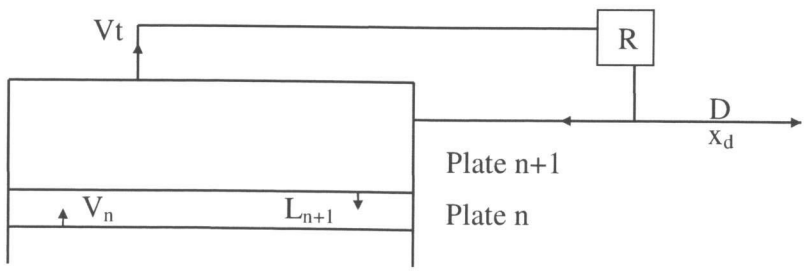
calibration graph for refractometer (produced in Experiment B)

equilibrium data for methyl*cyclo*hexane/toluene at atmospheric pressure.

Theory

Calculation of Number of Plates using the LEWIS-SOREL method

Material balance of top of column:



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

With respect to M.V.C. this becomes

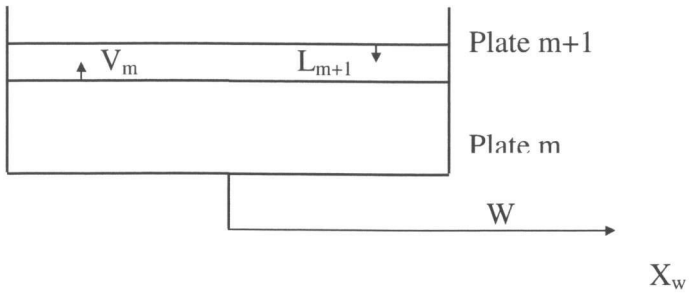
$y_n V_n = L_{n+1} x_{n+1} + D x_d$

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

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

$$y_n = \frac{L_{n+1}}{V_n} x_{n+1} + \frac{D}{V_n} x_d \qquad \text{..... (1)}$$

Material balance of bottom of column:



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

With respect to M.V.C. this becomes

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

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

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

$$y_m = \frac{L_m}{V_m} x_{m+1} - \frac{W}{V_m} x_w \qquad \text{..... (2)}$$

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

Sample Calculation:

Using a feed of a binary mixture of 60 mol percent methyl*cyclo*hexane and 40 mol percent toluene.

Top product required: 75 mol percent methyl*cyclo*hexane

Bottom product required: 44 mol percent methyl*cyclo*hexane

Reflux ratio 5:1

A material balance on the M.V.C. (most volatile component), methyl*cyclo*hexane gives,

Feed	=	Top product + Bottom product
100 x 0.60	=	0.75D + 0.44W
and D	=	100-W
100 x 0.60	=	0.75(100-W) + 0.44W
W	=	48.39 and D = 51.61

Also L_n = 5D (reflux ratio = 5)

L_n = 258.05

and $V_n = L_n + D$

V_n = 309.66

from equation (1)

$$y_n = \frac{258.05}{309.66} x_{n+1} + \frac{51.61 \times 0.75}{309.66}$$

$y_n = 0.83x_{n+1} + 0.13$ for the top section

$L_m = 258.05 + 100 = 358.05$

$V_m = L_m - W$

$= 358.05 - 48.39$

$V_m = 309.66 = V_n$

from equation (2)

$$y_m = \frac{358.05}{309.66} x_{m+1} + \frac{48.39 \times 0.44}{309.66}$$

$y_n = 1.16x_{m+1} - 0.07$ for the bottom section

From the equilibrium curve,

Composition on the top sieve plate is $y_t = 0.75$, $x_t = 0.72$

and $y_{t-1} = 0.83 \times 0.72 + 0.13 = 0.73$

x_{t-1} = 0.69

y_{t-2} = 0.83 x 0.67 + 0.13 = 0.69

x_{t-2} = 0.65

y_{t-3} = 0.83 x 0.65 + 0.13 = 0.67

x_{t-3} = 0.62

y_{t-4} = 0.83 x 0.62 + 0.13 = 0.65

x_{t-4} = 0.60

Since the feed composition is 0.60, it can be introduced on this tray (t-4).

y_{t-5} = 1.16 x 0.60 - 0.07 = 0.63

x_{t-5} = 0.58

y_{t-6} = 1.16 x 0.58 - 0.07 = 0.60

x_{t-6} = 0.54

y_{t-7} = 1.16 x 0.54 - 0.07 = 0.56

x_{t-7} = 0.50

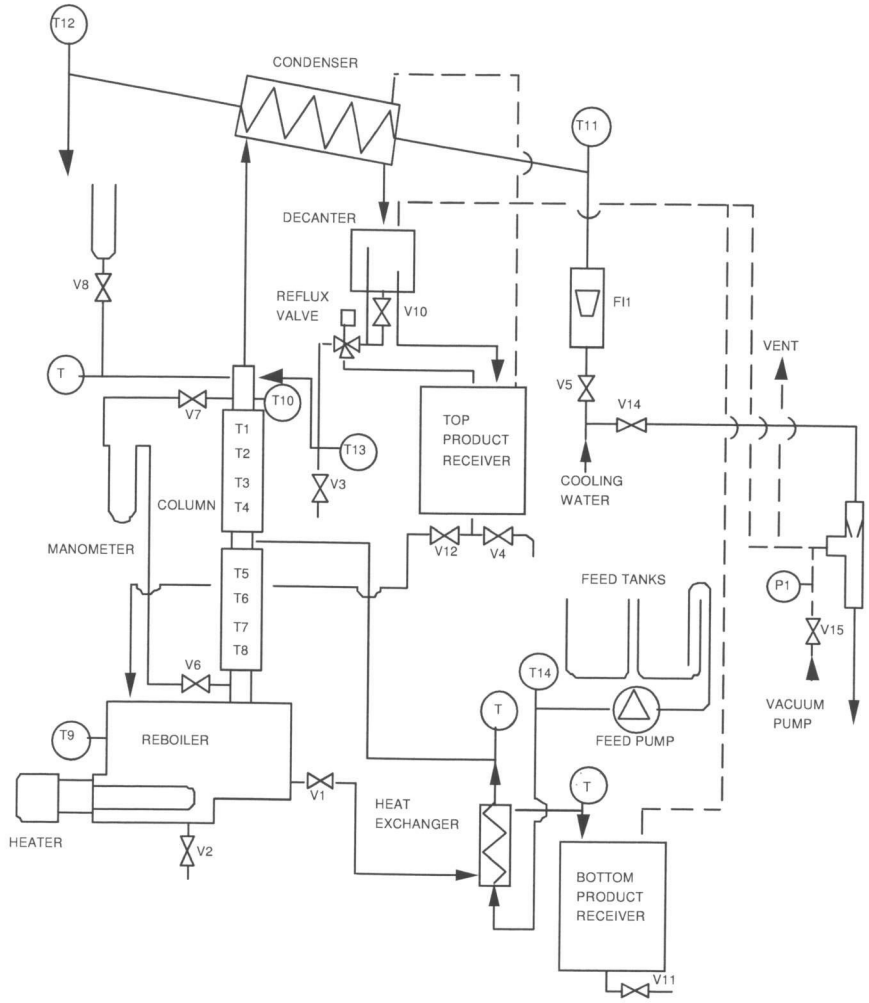
y_{t-8} = 1.16 x 0.50 - 0.07 = 0.51

x_{t-8} = 0.44

This composition is close to that required and can be withdrawn from the bottom product.

Theoretically, therefore, the distillation column containing eight sieve plates plus the boiler will give the compositions calculated above. However, as the experiment will show this is not in fact correct. Relate the theory and the results of the experiment with the previous experiment to determine column efficiency.

Equipment Set Up



Charge the feed bottle with 5 litres of a mixture of 65 mol percent methyl*cyclo*hexane and 35 mol percent toluene. Required for the mixture are:

1.17 litres of toluene

3.83 litres of methyl*cyclo*hexane

Charge the reboiler with 10 litres of a mixture of 25 mol percent methyl*cyclo*hexane and 75 mol percent toluene. Required for the mixture are:

7.15 litres of toluene

2.85 litres of methyl*cyclo*hexane

Make sure the filler cap on the top of the reboiler is firmly replaced.

Procedure

Turn on the power to the control panel.

Set the temperature selector switch to T9, the temperature in the reboiler, and open valve V5 admitting the cooling water to the condenser at a flow rate on FI1 of

approximately 3 litres/min. This rate may be varied according to the temperature of the water. If there is insufficient water flow, some of the vapour will be uncondensed and emerge from the vent pipe at the side of the unit in which case increase the water flow rate. If required, admit cooling water to the bottom product cooler. This is not always necessary as sufficient cooling takes place in the pipework to reduce the product temperature from the boiling point.

On the control panel turn the power controller for the reboiler heating element fully anti-clockwise and switch the switch turning on the power to the heating element to "power on" position. Another red lamp will illuminate indicating the heating element is on. Turn the power controller clockwise until a reading of approximately 1.5 kW is obtained on the digital wattmeter. The contents of the reboiler will begin to warm up and this can be observed on the temperature readout meter.

Eventually, vapour will begin to rise up the column and the progress of this can be clearly observed as well as detected by the increasing temperatures when switching the temperature selector on T8, T7, T6, T5, T4, T3, T2 and T1. Vapour will enter the condenser and reappear as droplets into the glass walled distillate receiver vessel. The distillate will build up a small level in the receiver and eventually overflow to the reflux regulator valve. Start the experiment with total reflux, meaning the condensed vapour will return to the column.

The cool distillate is then returning to the top of the column and will cascade down the trays forming a liquid level on the trays and bubbling of vapour passing through the liquid. The system will have reached an equilibrium condition when the temperatures T1, T2, T3, T4, T5, T6, T7 and T8 have reached an average steady temperature (but note cycling due to the intermittent reflux).

Before switching on the reflux switch, set the reflux ratio to 5:1, meaning 5 sec back to column and 1 sec to top product receiver, (see "Operational Procedures"). C- means back to column and C+ means to top product receiver. When this is done, switch the reflux valve on (on the control panel). You should now hear a click, meaning the reflux valve is working. You should also see condensed vapour flowing to the top product receiver.

The feed to the column must be admitted at the mid point (onto tray 5).

When the column has stabilised at total reflux (it takes 15 to 30 minutes), the flow of feed and the reflux can be started at the same time.

It is advisable to set a feed flow of 2 litres/hr (from the feed pump calibration graph).

As the flow into the column becomes established so more vapour will rise up the column and appear as condensate in the distillate receiver, allow this to flow to the top product receiver.

After feeding approximately 3 litres take a sample of the overheads through valve V3. When doing that, be careful never to drain the condensate return line ie. partially open valve V3 to leave a small amount of liquid in the line all the time. Take a further four samples.

Generally, when taking samples, drain a "discarding" sample of approximately 5 to 10 ml before taking the representative sample in a sample glass. Do not drain too much of the "discarding" sample because of the disturbance of the mass balance. Discard the "discarding" sample in a safe way. After the representative sample has been taken,

keep the sample glasses in an upright position. Do not overturn them because of the possibility of evaporation of the sample.

Record the refractive index for the taken overhead sample. In a similar manner **and preferably at the same time** take a sample of the bottom through valve V2.

CAUTION! THIS SAMPLE WILL BE HOT.

Record the refractive index for this sample, too.

Repeat the sample taking a further nine times during the experiment (before feed runs out).

Results

	Top Product Composition Mol Percent methylcyclohexane	**Bottom Product Composition Mol Percent methylcyclohexane**
1		
2		
3		
4		
5		
6		
7		
8		
9		
10		

EQUILIBRIUM DATA FOR METHYL*CYCLO*HEXANE/TOLUENE

x	t (C)	y
0	110.6	0
0.05		0.077
0.10	108.50	0.146
0.15		0.213
0.20	106.90	0.270
0.25		0.326
0.30	105.60	0.378
0.35		0.423

0.40	104.50	0.476
0.45		0.522
0.50	103.55	0.565
0.55		0.608
0.60	102.80	0.650
0.65		0.693
0.70	102.20	0.735
0.75		0.778
0.80	101.60	0.820
0.85		0.863
0.90	101.20	0.908
0.95		0.953
1.00	100.93	1.000

x = mole fraction of methyl*cyclo*hexane in liquid

y = mole fraction of methyl*cyclo*hexane in vapour

t = temperature, °C

7.9 Exercise F: Varying the Feed Position

Objective

To investigate the effect of varying the feed position under continuous operation.

Equipment Required

UOP3CC

65 mol % methyl*cyclo*hexane/ 35 mol % toluene

25 mol % methyl*cyclo*hexane/75 mol % toluene mixture

1 - 250 ml measuring cylinder graduated in mls.

1 - stop watch

1 - hand held refractometer

calibration curve for the feed pump

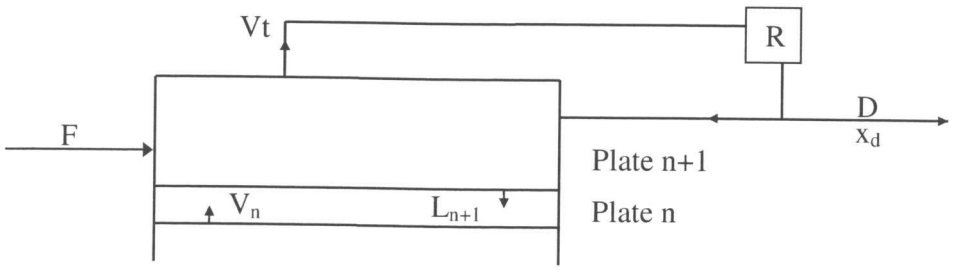
calibration graph for refractometer (produced in Experiment B)

equilibrium data for methyl*cyclo*hexane/toluene at atmospheric pressure.

Theory

The rig is provided with three feed positions, one above the top plate, one between the two four-plate sections (mid-point) and one below the bottom plate. In this way the rig can be run as a conventional distillation column (feed between the sections), as a rectifying column (feed below the bottom plate) or as a stripping column (feed above the top plate). As the rectifying case is closely approximated by the batch distillation done in Experiments A, C and D, the stripping case is chosen for this experiment.

Calculation of Number of plates using the Lewis-Sorel method



Material balance of top of the column

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

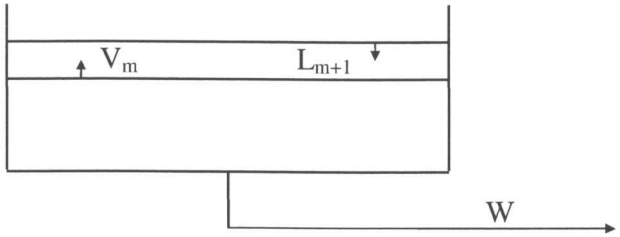
with respect to M.V.C. this becomes

$$V_n y_n = L_{n+1}x_{n+1} + Dx_D - Fx_F$$

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

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

$$y_n = \frac{L_n x_{n+1}}{V_n} + \frac{Dx_D}{V_n} - \frac{Fx_F}{V_n} \qquad \text{..... (1)}$$



Material balance of bottom of the column

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

with respect to M.V.C. this becomes

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

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

Since the liquid overflow is constant $L_m = L_{m-1}$

$$y_m = \frac{L_m x_{m+1}}{Vm_m} - \frac{Wx_B}{V_m}$$

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

Using a feed of a binary mixture of 63 mol percent methyl*cyclo*hexane and 37 mol percent toluene:

Top product required:	66 mol percent methyl*cyclo*hexane
Bottom product required:	21 mol percent methyl*cyclo*hexane
Reflux ratio	5:1

An overall material balance on the M.V.C. (most volatile component), methyl*cyclo*hexane gives,

Feed	=	Top product	+ Bottom product
100 x 0.63	=	0.66D	+ 0.21W
and D	=	100-W	
100 x 0.63	=	0.66(100-W)	+ 0.21W

$\underline{W = 6.67 \text{ and } D = 93.33}$

Also $L_n = 5D$ (reflux ratio = 5)

$\underline{L_n = 466.65}$

and $V_n = L_n + D - F = 466.65 + 93.33 - 100$

$\underline{V_n = 459.98}$

from equation (1)

$$y_n = \frac{466.65}{499.98} x_{n+1} + \frac{93.33 \times 0.66}{499.98} - \frac{100 \times 0.63}{499.98}$$

$\underline{y_n = 1.0.15x_{n+1} - 0.003}$

(for the top section and the bottom section of the column)

From the equilibrium curve, the composition on the top sieve plate is $y_t = 0.70$, $x_t = 0.66$

and	y_{t-1}	=	1.015 x 0.66 + 0.003 = 0.67
	x_{t-1}	=	0.62
	y_{t-2}	=	1.015 x 0.62 + 0.003 = 0.63
	x_{t-2}	=	0.58
	y_{t-3}	=	1.015 x 0.58 + 0.003 = 0.59
	x_{t-3}	=	0.55
	y_{t-4}	=	1.015 x 0.58 + 0.003 = 0.56
	x_{t-4}	=	0.50
	y_{t-5}	=	1.015 x 0.50 - 0.003 = 0.50
	x_{t-5}	=	0.43
	y_{t-6}	=	1.015 x 0.43 - 0.003 = 0.43
	x_{t-6}	=	0.35

y_{t-7} = 1.015 x 0.35 - 0.003 = 0.35

x_{t-7} = 0.28

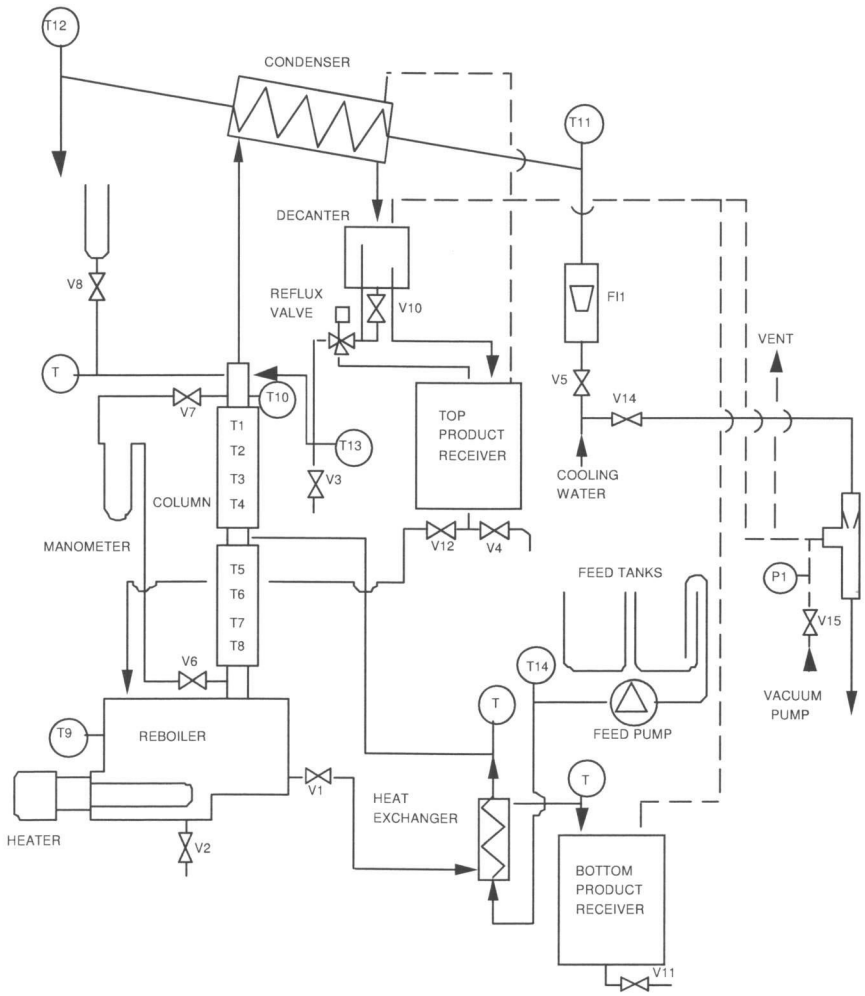
y_{t-8} = 1.015 x 0.28 - 0.003 = 0.28

x_{t-8} = 0.21

This composition is close to that required and can be withdrawn from the bottom product.

Theoretically, therefore, the distillation column containing eight sieve plates plus the boiler will give the compositions calculated above. However, as the experiment will show this is not in fact correct. Relate the theory and the results of the experiment with the previous experiment to determine column efficiency.

Equipment Set Up



Charge the feed beaker with 5 litres of a mixture of 63 mol percent methyl*cyclo*hexane and 37 mol percent toluene. (Required for the mixture are 1.65 litres of toluene and 3.35 litres of methyl*cyclo*hexane).

Charge the reboiler with sufficient mixture of 25 mol percent methyl*cyclo*hexane and 75 mol percent toluene to overflow valve (V1).

Make sure the filler cap on the top of the reboiler is firmly replaced.

Procedure

Turn on the power to the control panel.

Set the temperature selector switch to T9, the temperature in the reboiler and open valve V5 admitting the cooling water to the condenser at a flow rate of FI1 of approximately 3 litres/min. This rate may be varied according to the temperature of the water. If there is insufficient water flow, some of the vapour will be uncondensed and emerge from the vent pipe at the side of the unit in which case increase the water flow rate. If required, admit cooling water to the bottom product cooler. This is not always necessary as sufficient cooling takes place in the pipework to reduce the product temperature from the boiling point.

On the control panel turn the power controller for the reboiler heating element fully anti-clockwise and switch the switch turning on the power to the heating element to "power on" position. Another red lamp will illuminate indicating the heating element is on.

Turn the power controller clockwise until a reading of approximately 1.5 kW is obtained on the digital wattmeter. The contents of the reboiler will begin to warm up and this can be observed on the temperature readout meter.

Eventually, vapour will begin to rise up the column and the progress of this can be clearly observed as well as detected by the increasing temperatures when switching the temperature selector on T8, T7, T6, T5, T4, T3, T2 and T1. Vapour will enter the condenser and reappear as droplets into the glass walled distillate receiver vessel. The distillate will build up a small level in the receiver and eventually overflow to the reflux regulator valve. Start the experiment with total reflux, meaning the condensed vapour will return to the column.

The cool distillate is then returning to the top of the column and will cascade down the trays forming a liquid level on the trays and bubbling of vapour passing through the liquid. The system will have reached an equilibrium condition when the temperatures T1, T2, T3, T4, T5, T6, T7 and T8 have reached an average steady temperature (but note cycling due to the intermittent reflux).

Before switching on the reflux switch, set the reflux ratio to 5:1, meaning 5 sec back to column and 1 sec to top product receiver, (see "Operation"). C- means back to column and C+ means to top product receiver. When this is done, switch the reflux valve on (on the control panel). You should now hear a click, meaning the reflux valve is working. You should also see condensed vapour flowing to the top product receiver.

The feed to the column must be admitted above the top plate.

When the column has stabilised at total reflux (it takes 15 to 30 min.), the flow of feed and the reflux can be started at the same time.

It is advisable to set a feed flow of 2 litres/hr initially (from the feed pump calibration graph).

As the flow into the column becomes established so more vapour will rise up the column and appear as condensate in the distillate receiver, allow this to reach the overflow and flow to the top product receiver.

After feeding approximately 3 litres take a sample of the overheads through valve V3. When doing that, be careful never to drain the condensate return line ie. partially open valve V3 to leave a small amount of liquid in the line all the time.

Generally, when taking samples, drain a "discarding" sample of approximately 5 to 10 ml before taking the representative sample in a sample glass. Do not drain too much of the "discarding" sample because of the disturbance of the mass balance. Discard the "discarding" sample in a safe way. After the representative sample has been taken, keep the sample glasses in an upright position. Do not overturn them because of the possibility of evaporation of the sample.

Record the refractive index for the taken overhead sample. In a similar manner **and preferably at the same time** take a sample of the bottom through valve V2.

CAUTION! THIS SAMPLE IS HOT.

Record the refractive index for this sample, too.

Repeat the sample taking a further nine times during the experiment (before feed runs out).

Results

	Top Product Composition Mol Percent methylcyclohexane	**Bottom Product Composition Mol Percent methylcyclohexane**
1		
2		
3		
4		
5		
6		
7		
8		
9		
10		

Conclusion

The results from previous exercises (A, C and/or D) exercise, as results for a rectifying column (feed below the bottom plate) should be compared to the results from this exercise for a stripping column (feed above the top plate).

7.10 Exercise G: Using a PID Controller

Objectives

To demonstrate a typical application of a PID controller, to observe the response of the process to a change in set point and to a disturbance, and to adjust the controller setting for optimum process control.

Method

To configure two alternative control loops using the UOP3CC in conjunction with the PCT20H PID controller: Reboiler power control from column temperature T7 or Reflux ratio control from top tray temperature T1.

Equipment Required

UOP3CC

PCT20H PID controller*

(*Any PID controller with input and output signals conditioned to 0-5 Vdc may be used if the PCT20H is not available. However, Armfield cannot supply instructions for controllers not supplied by Armfield)

Theory

'**PID**' is an acronym for **P**roportional **I**ntegral **D**erivative.

In proportional control, the process variable sensor (for example, a temperature sensor) sends a signal to the controller that varies with the value of the process variable. The controller sends a signal to the controlled parameter (for example, the power to a heater) that is proportional to the signal from the sensor. In some cases the controlled parameter can only be off or on, in which instance the controller varies the time for which the parameter is switched on over a particular **cycle time**.

With proportional control action alone, the controller produces a signal that is proportional to the error (the difference between the monitored variable and the set point value). This creates an offset between setpoint value and actual value (the controller only supplies an output when there is an error, so there is no controller output when the value is not at the set point). It also generates an overshoot (the system will oscillate above and below the setpoint value at the start of the control period until stability is attained).

With integral control action, the controller gives an output that is proportional to the time integral of the error. Integral control action can potentially be used alone to control a process, but is normally used in conjunction with proportional action. When used with proportional action it can eliminate offset. It can also cause higher maximum deviation and a longer response time than with proportional action alone.

With derivative control action, the controller gives an output that is proportional to the derivative of the rate of change of the error. The output is related only to the rate of

change, not to the magnitude of the error. Derivative control action cannot be used alone, but must be combined with another action such as proportional control action. When used with proportional action, derivative control can eliminate excessive oscillation. It cannot eliminate offset errors inherent in proportional action

Proportional, integral and derivative control actions are combined to eliminate offset, reduce maximum deviation and minimise the frequency of oscillation. Finding the optimum values of the proportional band (P), integral time (I) and derivative time (D) for a particular process is often referred to as **tuning** or **optimisation**.

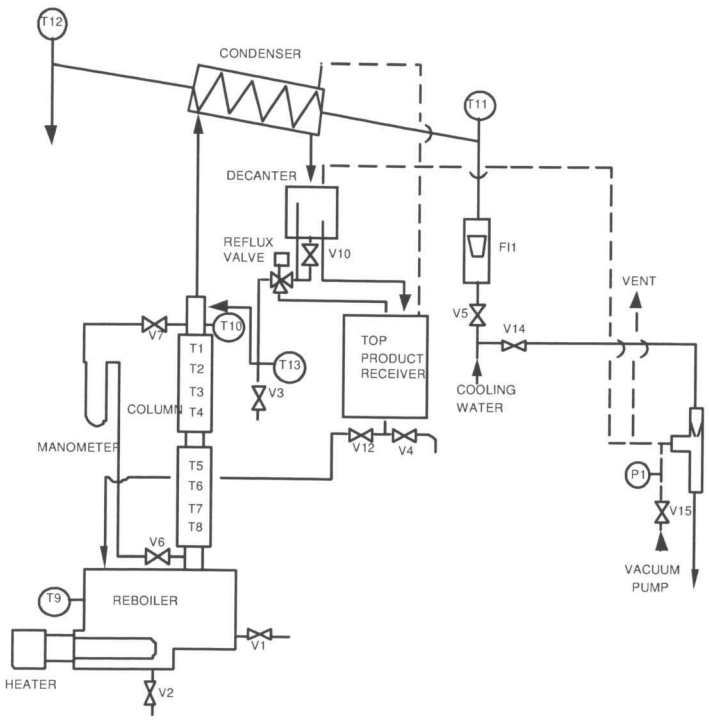
In this exercise a PID controller will be used to investigate two different control loops, one involving power control and one involving fluid ratio control. In both instances the process variable monitored by the controller is temperature.

Equipment Set Up

Make up 10 litres of a mixture of 50 mol percent methyl*cyclo*hexane and 50 mol percent toluene. Required for the mixture are:

4.57 litres of toluene

5.43 litres of methyl*cyclo*hexane



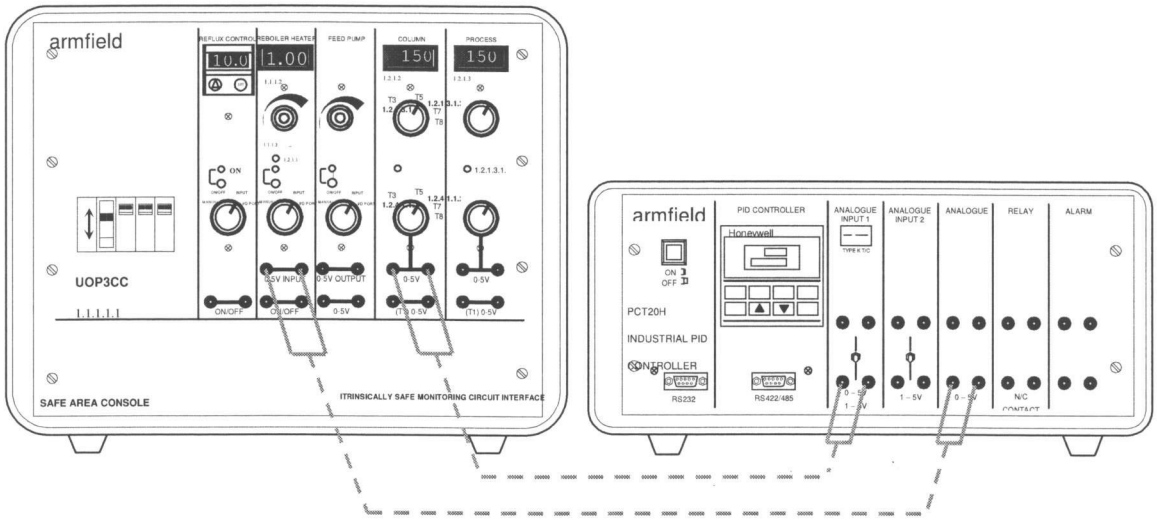
Set the equipment to operate at total reflux by switching off the reflux ratio timer on the console.

Load the 10 litre charge of prepared feed mixture (the liquid to be distilled) directly into the reboiler through the filler cap provided. Make sure the filler cap on the top of the reboiler is firmly replaced. At total reflux there will be no feed or top product or bottom product.

Setup for reboiler power control:

Note: The following instructions assume the use of the PCT20H. If using your own controller then make adjustments to the procedure as required.

Connect the PCT20H process controller to the UOP3CC Electrical Console using leads terminated with banana plugs. Analog connections are made using leads with red & black plugs. Match the colours on the sockets of the PCT20H and the UOP3CC and make the connections as follows:



If a computer with Armfield software is available then the response of the process under PID control can be monitored and logged using the Data Logging screen. Refer to experimental sheet J for details on connecting a PC to UOP3CC.

Refer to the PCT20H or controller manufacturer's Instruction Manuals for details on how to configure and operate the process controller.

Set the Analog Input 1 selector switch to 0-5V on the PCT20H.

Set the Reboiler Power Control selector switch to the 'INPUT SOCKET' position on UOP3CC.

Set the temperature selector switch to T7 on UOP3CC.

Set the Reflux Control and Feed Pump Control selector switches to the 'MANUAL' position on UOP3CC.

To ensure correct configuration of the process controller, the controller should be set to the default configuration (UOP3CC Reboiler power control), as described in the PCT20H Instruction Manual, before changing specific settings as required for this demonstration. Configuration of the PCT20H process controller can be changed using software via a PC or via the buttons on the front of the controller. Note that when changing settings by these buttons some options may not be displayed in the menu until other options have been set.

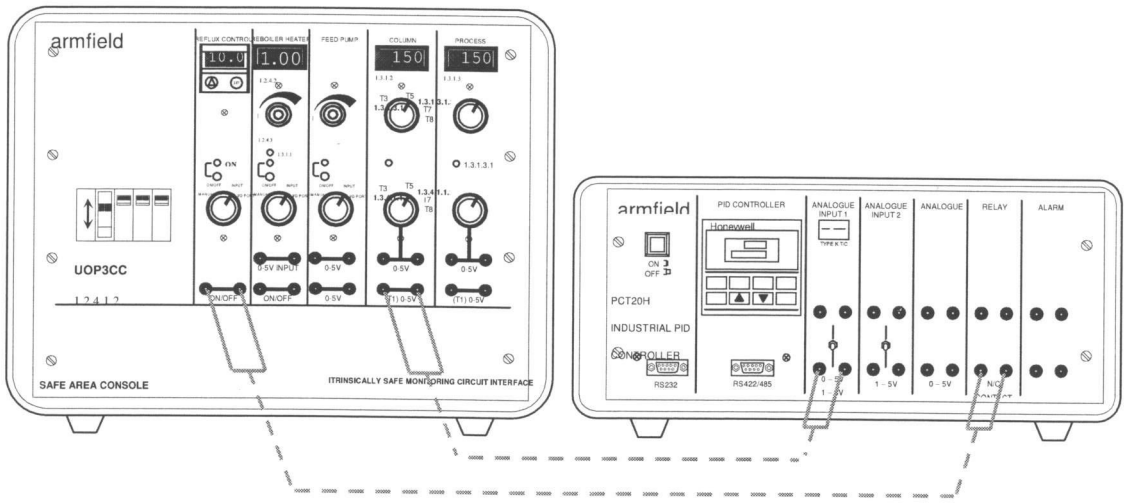
If using a controller other than PCT20H then the important settings are as follows:

Input signal	0 – 5 V
Corresponding input range	0 - 150°C
Output signal proportional	0-5 Vdc
Control algorithm	PID
Control action	Reverse
Initial Proportional Band (P)	50%
Initial Integral action (I)	4 RPM
Initial Derivative action (D)	0

Setup for reflux control:

Note: The following instructions assume the use of the PCT20H. If using your own controller then make adjustments to the procedure as required.

Connect the PCT20H process controller to the UOP3CC Electrical Console using leads terminated with banana plugs. Analog connections are made using leads with red & black plugs, digital connections are made using leads with yellow plugs. Match the colours on the sockets of the PCT20H and the UOP3CC and make the connections as follows:



Set the Analog Input 1 selector switch to 0-5V on PCT20H.

Set the Reflux Control selector switch to the 'INPUT SOCKET' position on UOP3CC.

Set the temperature selector switch to T1 on UOP3CC.

Set the Reboiler Power Control and Feed Pump Control selector switches to the 'MANUAL' position on UOP3CC.

To ensure correct configuration of the process controller, the controller should be set to the default configuration (UOP3CC Reflux control), as described in the PCT20H

Instruction Manual, before changing specific settings as required for this demonstration. Configuration of the PCT20H process controller can be changed using software via a PC or via the buttons on the front of the controller. Note that when changing settings by these buttons some options may not be displayed in the menu until other options have been set.

If using a controller other than PCT20H then the important settings are as follows:

Input signal	0 – 5 V
Corresponding input range	0 - 150°C
Output signal proportional	0-5 Vdc
Control algorithm	PID
Control action	Reverse
Initial Proportional Band (P)	50%
Initial Integral action (I)	4 RPM
Initial Derivative action (D)	0

Procedure

Refer to the PCT20H manual if you need further information on operating the PID controller beyond that covered in this exercise.

Reboiler power control:

Adjust the Setpoint to the required value for T7.

The controller will start in manual operation with an output of 0% (No power to the reboiler). Adjust the manual output from the controller until the tray temperature T7 has stabilised at the required value then select AUTO to allow the controller to maintain T7.

An investigation may now be made to establish the effect of set point changes and disturbances, and to optimise the P(PROP BD), I(RATE MIN) and D(RSET RPM) settings.

To investigate the effect of a set point change, take a sample, then increase the set point setting by a significant amount (e.g. 20 °C), then take further samples at regular intervals until the process has stabilised once more. This should be combined with observation of the appearance of the trays. Change the set point again, this time a decrease of the same amount as the initial increase (e.g. 20 °C). Take samples and make observations as before.

A disturbance to the system may be produced by introducing feed of a different concentration.

The P, I and D parameters may be adjusted on the controller as described in the PCT20H manual. Alter each parameter separately and investigate the effect of the change.

Optimisation:

Optimum values for P, I and D can be determined by monitoring the response of the system following a small disturbance using standard techniques. However, the controller used in PCT20H includes an auto-tune ("Accutune") facility which may be used to optimise the PID settings automatically.

Accutune is only available when the controller is in automatic mode (i.e. when the controller is controlling the process).

Reset the PROP BD, RATE and RSET controller settings to the values provided in the set-up section of this exercise before activating auto-tune.

To activate Accutune; enable Accutune in the Setup menu then press the Lower Display key until Tune appears then press the 'Δ' key until 'T' appears at the left hand side of the upper display. This disappears when tuning is complete.

Before activating Accutune, Fuzzy overshoot suppression can be enabled in the Setup menu if this function is required.

Make a note of the new PROP BD, RATE and RSET values after tuning.

Refer to the manufacturers instruction manual supplied with the process controller for more information regarding use of the Accutune facility.

Reflux control:

An investigation may now be made to establish the effect of set point changes, disturbances, and changes to the P(PROP BD), I(RATE MIN), D(RSET RPM), and cycle time (CYC SEC) settings.

To investigate the effect of a set point change, take a sample, then increase the set point setting by a significant amount (e.g. 20 °C), then take further samples at regular intervals until the process has stabilised once more. This should be combined with observation of the appearance of the trays. Change the set point again, this time a decrease of the same amount as the initial increase (e.g. 20 °C). Take samples and make observations as before.

A disturbance to the system may be produced by introducing feed of a different concentration.

The P, I, D and cycle time parameters may be adjusted on the controller as described in the PCT20H manual. Alter each parameter separately and investigate the effect of the change.

NOTE: The output range indicated on the lower display of the controller is 0-50 corresponding to an actual output of 0-100%.

i.e. For 0% output (Total Reflux) set controller output to 0

For 50% output (Reflux Ratio 1:1) set controller output to 25

For 100% output (no Reflux) set controller output to 50.

Optimisation:

This is described above in the procedure for reboiler power control.

Results

Samples may be taken and concentration measured using a hand-held refractometer and the calibration graph obtained in Exercise B. Graphs of the process variable and the control parameter against time, for each controller setting, may be informative.

7.11 Exercise H: Using a Programmable Logic Controller

Objective

To demonstrate a typical application of a Programmable Logic Controller (PLC) with analog inputs & outputs. To observe the response of the process to a change in setpoint or disturbances. To adjust the settings of the PLC for optimum control of the process.

Method

To use the UOP3CC with a programmable logic controller, PCT19BR, and a compatible PC to monitor and adjust the settings of the PID blocks in the logic controller ladder program.

Equipment Required

UOP3CC

4.57 litres of toluene

5.43 litres of methyl*cyclo*hexane

PCT19BR with supplied leads and cables*

PC running Windows™ 98 or later with an available 9-pin serial interface port

(*Any PLC can be used for the demonstration provided that the input and output signals are conditioned to 0-5 Vdc. However, Armfield cannot supply instructions for using any PLC not supplied by Armfield)

Theory

A programmable logic controller is a device that can be programmed to perform control functions. They are used in many situations and applications, such as controlling production processes in manufacturing facilities. They are easy to install, program test and troubleshoot. They can be used in a network environment while remaining secure, and they have rapid response for applications requiring high-speed control.

The UOP3CC Continuous Distillation Column can be used for single loop process control demonstrations using a Programmable Logic Controller (PCT19BR). It is possible to configure 2 different control loops individually:

1) Reboiler Power Control from a column temperature (T7)

2) Reflux Control from the top tray temperature (T1)

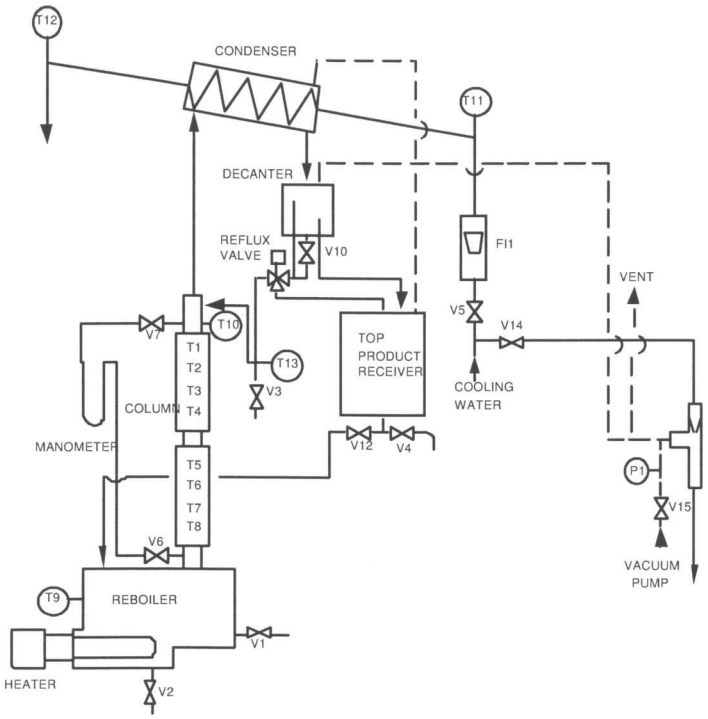
Either loop may be simply configured by connecting the PLC (PCT19BR) to the UOP3CC Electrical Console as described in the Set Up section of this experiment. The PCT19BR PLC is programmed by uploading a ladder program from a compatible PC (it need not be connected to the PC during operation, only while uploading the control program required). The procedure for doing so is fully described in the PCT19BR manual.

Equipment Set Up

Make up 10 litres of a mixture of 50 mol percent methyl*cyclo*hexane and 50 mol percent toluene. Required for the mixture are:

4.57 litres of toluene

5.43 litres of methyl*cyclo*hexane



Set the equipment to operate at total reflux by switching off the reflux ratio timer on the console.

Load the 10 litre charge of prepared feed mixture (the liquid to be distilled) directly into the reboiler through the filler cap provided. Make sure the filler cap on the top of the reboiler is firmly replaced. At total reflux there will be no feed or top product or bottom product.

Refer to the PCT19BR Instruction Manual for details on how to monitor and adjust the configuration of the PLC.

The response of the process under PID control can be monitored and logged using the Armfield software supplied with the UOP3CC. Ensure that the UOP3CC console is connected to the PC as described on page 21.

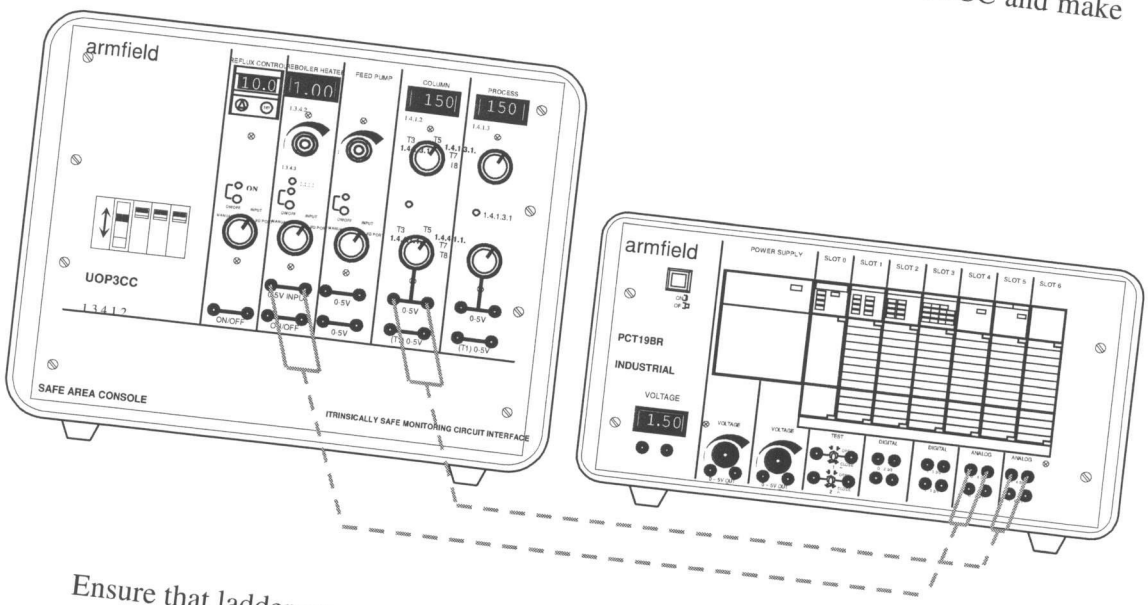
Reboiler Power Control:

Set the Reboiler Power Control selector switch to the 'INPUT SOCKET' position.

Set the temperature selector switch to T7.

Set the Reflux Control selector switch to the 'MANUAL' position.

Connect the PCT19BR PLC to the UOP3CC Electrical Console using leads terminated with banana plugs. Analog connections are made using red & black ended connecting leads, digital connections are made using yellow ended leads. Match the colours on the sockets of the PCT90BR and the UOP3CC and make the connections as follows:



Ensure that ladder program PCT19BR has been downloaded to the PLC.

Set the following values in PID block PID1 (proportional analog output):

P	50%
I	4
D	0

Press F1 to select manual output.

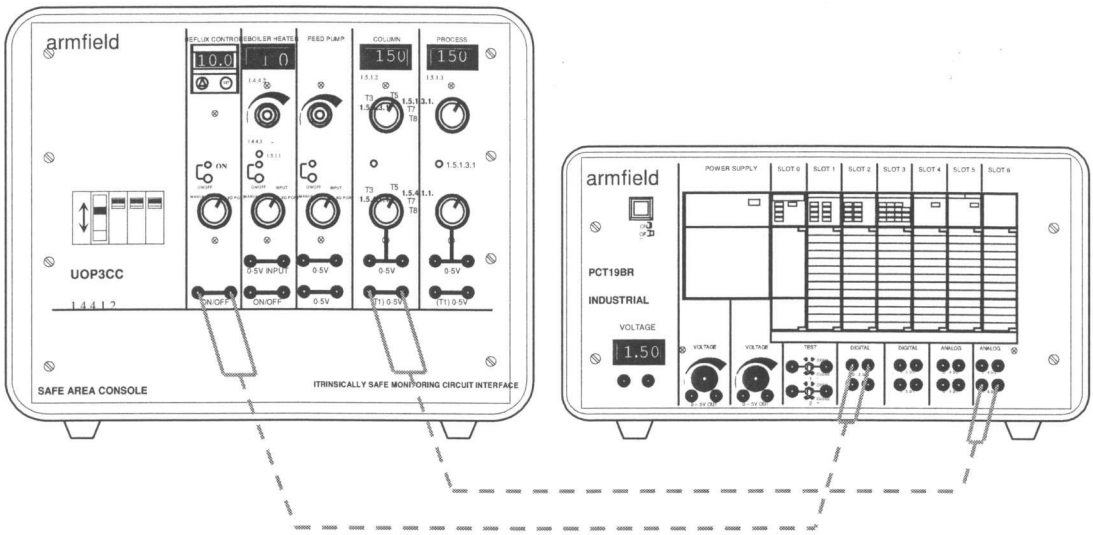
Reflux control:

Set the Reflux Control selector switch to the 'INPUT SOCKET' position.

Set the temperature selector switch to T1.

Set the Reboiler Power Control selector switch to the 'MANUAL' position.

Connect the PCT19BR PLC to the UOP3CC Electrical Console using leads terminated with banana plugs. Analog connections are made using red & black ended connecting leads, digital connections are made using yellow ended leads. Match the colours on the sockets of the PCT90BR and the UOP3CC and make the connections as follows:



Ensure that ladder program PCT19BR has been downloaded to the PLC. The following values should be initially set in PID block PID2 (proportional analog output):

P	35%
I	2
D	0

Press F1 to select manual output.

Procedure

Reboiler power control:

Adjust the Setpoint to the required value for T7.

Adjust the manual output from the controller until the tray temperature T7 has stabilised at the required value then press F1 to switch to AUTO to allow the controller to maintain T7.

An investigation may be made to establish the effect of disturbance and to optimise the P, I and D settings.

To investigate the effect of a set point change, take a sample, then increase the set point setting by a significant amount (e.g. 20 °C), then take further samples at regular intervals until the process has stabilised once more. This should be combined with observation of the appearance of the trays. Change the set point again, this time a decrease of the same amount as the initial increase (e.g. 20 °C). Take samples and make observations as before.

A disturbance to the system may be produced by introducing feed of a different concentration.

The P, I and D parameters may be adjusted on the controller as described in the PCT19BR manual. Alter each parameter separately and investigate the effect of the change. After establishing the effect of each control parameter used alone,

select the best setting for the proportional band P, then adjust the I and D settings to give optimum performance.

Reflux control:

Adjust the Setpoint to the required value for T1.

Adjust the manual output from the controller until the tray temperature T1 has stabilised at the required value then press F1 to switch to AUTO to allow the controller to maintain T1.

An investigation may now be made to establish the effect of disturbance and to optimise the P, I and D settings.

To investigate the effect of a set point change, take a sample, then increase the set point setting by a significant amount (e.g. 20 °C), then take further samples at regular intervals until the process has stabilised once more. This should be combined with observation of the appearance of the trays. Change the set point again, this time a decrease of the same amount as the initial increase (e.g. 20 °C). Take samples and make observations as before.

A disturbance to the system may be produced by introducing feed of a different concentration.

The P, I and D parameters may be adjusted on the controller as described in the PCT19BR manual. Alter each parameter separately and investigate the effect of the change. After establishing the effect of each control parameter used alone, select the best setting for the proportional band P, then adjust the I and D settings to give optimum performance.

Further laboratory work is possible creating new ladder logic programs and uploading these to the PLC for testing. RS Logix software is provided with the PCT19BR for creating new ladders. This is described in the PCT19BR manual.

Results

Tabulate your results in an appropriate manner. Include full information on the PID settings, set point values, the RI values from the samples taken and the times at which those samples were taken, and the results of visual observation of the column.

7.12 Exercise I: Using Software

Objective

To use PC control and data logging software with the UOP3CC.

Method

To study two different control loops using the UOP3CC with Armfield data logging software. To demonstrate a typical application of a PC for PID process control, to observe the response of the process to a change in setpoint or disturbances, and to adjust the settings of the controller for optimum control of the process.

Equipment Required

UOP3CC with supplied USB cable

4.57 litres of toluene

5.43 litres of methyl*cyclo*hexane

PC running Windows™ 98 or later with Armfield UOP3CC software installed

Theory

Software control and monitoring of processes from a PC may be used in situations when dedicated process controllers are not required. Computer interfaces vary considerably. The Armfield software is representative of the basic elements found in most process control software, providing visual display and logging of sensor outputs and both manual and automated control of the equipment. The software includes configurable PID control for two different control loops:

Reboiler Power Control from a column temperature (T7)
Reflux Control from the top tray temperature (T1)

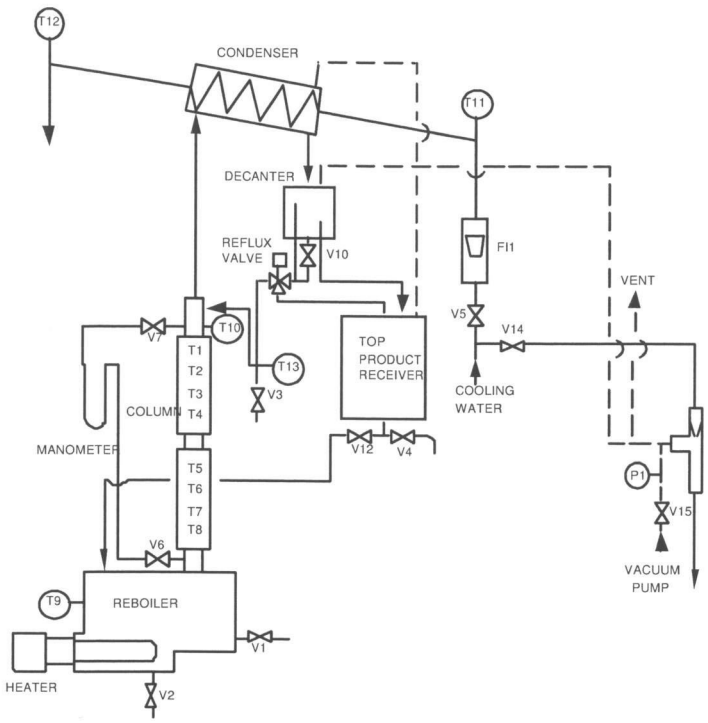
Equipment Set Up

Ensure that the software has been installed as described in the Installation Guide (page i at the back of this manual).

Make up 10 litres of a mixture of 50 mol percent methyl*cyclo*hexane and 50 mol percent toluene. Required for the mixture are:

4.57 litres of toluene

5.43 litres of methyl*cyclo*hexane



Set the equipment to operate at total reflux by switching off the reflux ratio timer on the console.

Load the 10 litre charge of prepared feed mixture (the liquid to be distilled) directly into the reboiler through the filler cap provided. Make sure the filler cap on the top of the reboiler is firmly replaced. At total reflux there will be no feed or top product or bottom product.

Refer to the PCT19BR Instruction Manual for details on how to monitor and adjust the configuration of the PLC.

The response of the process under PID control can be monitored and logged using the Armfield software supplied with the UOP3CC. Ensure that the UOP3CC console is connected to the PC as described on page 21.

Procedure

Reboiler Power Control

Ensure that the Reboiler Power Control selector switch is set to the 'I/O PORT' position.

Ensure that the Reflux Control and Feed Pump Control selector switches are set to the 'MANUAL' position.

Click on the PID button for the Reboiler Controller from the mimic diagram screen in the Armfield software.

The controller settings for this control demonstration are tabulated below.

P	50%	
I	15	seconds
D	0	seconds

Adjust the manual output from the Controller until tray temperature T7 has stabilised at the required value, then select AUTO to allow the controller to maintain T7. Experiments may now be performed to establish the effect of disturbances and to optimise the controller settings.

Reflux Control

Ensure that the Reflux Control selector switch is set to the 'I/O Port' position.

Ensure that the Reboiler Power Control and Feed Pump Control selector switches are set to the 'MANUAL' position.

Click on PID2 from the mimic diagram of the UOP3CC software.

Enter typical values on the Reflux Controller Screen, e.g.

Cycle time 10 secs

Initial ratio 4:1

Select Manual Control, click 'Apply', and allow the column to stabilise.

Enter typical values on the Reflux Controller Screen, e.g.

Cycle time 10 secs

Initial ratio 4:1

Proportional Band 50%

Set Point (T1) to suit binary mixture

Select Automatic Control, click 'Apply', and allow the controller to maintain T1.

Experiments may now be performed to establish the effect of disturbances and to optimise the controller settings.

8 Installation Guide

Before operating the equipment, it must be unpacked, assembled and installed as described in this Installation Guide. Safe use of the equipment depends on following the correct installation procedure.

Remove the equipment from the packaging. The distillation process unit is packed laying flat with the control console connected by 3.5m of cable which is designed to allow the 'non-flameproof' console to be sited more than 2.0m away from the process. Lift the unit to the vertical position, supported on the adjustable feet. The frame includes cross - members so that a fork lift, pallet truck or other lifting device can be used to transport it in the upright position.

The equipment is supplied with the control console inter-connected to the distillation column unit by the appropriate cables.

Sufficient cable is supplied to allow positioning of the console in front of the process unit and outside the 2.0m Zone 2 area around it.

In some instances it may be necessary to install the console in a separate room. This involves the disconnection of the gland plates and connectors at the rear of the console and their re-connection after the cables have been passed through the wall or partition. This procedure must be carried out by a competent electrician and extreme care is required in ensuring the individual cables are reconnected in exactly the same positions. Failure to do this will compromise the safety features built in to the console.

The location where the cable passes through the partition must be fully sealed to prevent flammable vapour in the hazardous area from escaping to the non-hazardous area.

The control console is designed to be bench mounted and should be positioned on a suitable firm, level surface.

Feed Tanks

The glass feed tanks/bottles (5) and (6) are packed separately and require to be inserted into the retaining rings adjacent to the feed pump (7).

Feed Tank Lids

One of the feed tank lids/caps incorporates a suction pipe to which a length of Viton rubber tubing is attached. The free end of this tubing must be attached to the temperature sensor (T14) at the union located to the left of the pump head.

Feed Pump

The peristaltic feed pump (7) has a pivoting clamp on the pumphead for fitting the flexible tubing. Before installing the flexible tubing ensure that the adjusters on either side of the pumphead (at the bottom) have been set to the diameter of tube in use. (The standard tubing supplied has a bore of 3.2mm so the indicator must align with the 3.2

mark when the head is unclamped.) To install or replace the flexible tubing pivot the clamp (on the top of the pumphead) upwards and backwards to expose the rotor. Load the tubing into the slot between the rotor and pumphead then close the pumphead by pivoting the clamp forwards and downwards. The clamp will click shut. The tube will retain its elasticity for many hours of use but there is sufficient length to allow the tube to be moved to other positions of less wear. It is essential to release the clamp when the equipment is not being used to prevent permanent deformation of the tube.

Low Level Switch

The low level switch (17) must be installed in the reboiler in the boss provided. After tightening the switch, the signal cable must be connected by pushing the plug firmly into the socket on top of the switch.

Decanter Vessel (phase separator)

The decanter vessel (11) is removed from the equipment to prevent damage during transit and must be reinstalled in the process pipework as follows:

Locate the decanter vessel below the outlet pipe at the bottom of the condenser, slide the vessel onto the outlet pipe then tighten the coupling. When correctly installed the outlet pipe should protrude 25mm inside the top of the decanter vessel and valve V10 should face forwards.

Attach the flexible vent pipework (tee fitting) to the vent outlet pipe on the top of the decanter vessel then tighten the coupling.

Connect the flexible tube from the tapping on the top of the receiver vessel to the connection at the base of the adjustable overflow in the decanter vessel then tighten the coupling.

Connect the flexible tube from the inlet to the reflux solenoid valve to the tapping at the rear of the horizontal manifold below the decanter vessel then tighten the coupling.

Ensure that valve V10 is open to allow normal operation of the reflux system.

Bund Tray

The stainless steel bund tray must be positioned between the four feet of the frame, on the floor.

Bottom Product Cooler/Feed Preheater

The bottom product cooler (15) is mounted directly to the compression union (16) on top of the bottom product tank (9). The flexible pipe from the reboiler overflow valve (V1) is connected to the inlet of the heat exchanger by a similar union (14). Facility is available for passing feed from the feed pump through this heat exchanger in order to preheat the feed before entering the distillation column. Alternatively, a cooling water supply can be connected (at the base of the heat exchanger) and the outlet guided to drain.

Vent

If operation at atmospheric pressure is required the 4.0m length of tubing supplied must be attached to the free end of the vent pipe from the common connection on the top product receiver, and positioned in a fume cupboard or suitable atmospheric vent outlet. If operation at reduced pressures is required the vent pipe is simply connected to the vacuum pump (20) suction at connector (23).

Dosing Vessel.

Used for azeotropic distillation experiments. The dosing unit (24) must be fixed in position (see page 10) with the screws provided and the PTFE tube connected to the fitting (C) at the top of the column.

Cold Water

The cold water supply inlet connection (19) to the condenser is situated at the base of the flowmeter on the right-hand side of the equipment.

Connect the hose nozzle (19) to a supply of cold water of 15.0 litres/minute at 2 bar (absolute) using 12mm I.D. reinforced flexible hose (not supplied). Secure the connections using suitable pipe clips.

Drain (Warm Water)

The condenser drain pipe is situated at the rear of the equipment and terminates in a hose nozzle (22). Connect the hose nozzle to a suitable drain using 12mm I.D. reinforced flexible tubing (not supplied). Secure the connections using pipe clips.

Drain (Vacuum pump water)

Motive water passing out of the vacuum pump must be routed to a suitable drain using flexible hose (not supplied). During reduced pressure operation a trace of solvent may be condensed into the motive water. If local regulations do not allow even very small quantities of solvent to be discharged to drain, the water from the vacuum pump must pass through a simple separator vessel before the drain.

Solvent Vapour Extraction

All solvent vessels and pipework are connected to a common vent pipe at the right-hand rear of the equipment (attached to the common connection on the top product receiver). It is recommended that, for operation under atmospheric pressure the flexible PTFE tubing provided be connected to this and routed to a safe place outside the building or to a fume cupboard so that any vapours produced by abnormal conditions will be dispersed safely. For operation under reduced pressure conditions, the vent pipe must be connected directly to the inlet of the vacuum pump at (23).

Solvent Spillage

A stainless steel bund tray is provided which must be placed directly beneath the process unit on the floor, between the four support legs. This is designed to contain any accidental spillage within the confines of the framework and thus within the Zone 1 area. Any accidental spillage must be immediately dealt with.

Switch on electrical power to the control console by moving the RCD switch to the UP position. The LOW LEVEL lamp in the reboiler heater section of the console will be illuminated. The reboiler power, reflux timer, column temperature and process temperature digital display will also be illuminated.

Ensure that all valves (V1 to V15) are in the closed position (see flow diagram). Valve numbers are not consecutive. Valves included are V1, V2, V3, V4, V5, V6, V7, V10, V12, V14 and V15. Ensure that the insulated jacket is not positioned around the shell of the condenser. Remove it if it has been fitted.

Remove the filler plug (21) from the top of the reboiler and, using a suitable funnel, fill the reboiler with 10 litres of water.

The LOW LEVEL lamp will go out. The reboiler level switch operation should be tested by draining water from the reboiler using drain valve (V2) and refilling through the filler cap.

Turn on laboratory cold water supply. Open flow control valve (V5) to give maximum flow into the condenser (8) via flowmeter (FI1). Check for leaks.

To prime the U-tube manometer (ΔP1), disconnect the black Viton tubing at the valves (V6) and (V7). Using a suitable syringe (not supplied) filled with clean water, inject the water into the manometer. It is advisable to hold one end of the Viton tubing of valve V7 vertically, insert the syringe and very slowly inject the water so that it flows down the inside wall of the tube. This will prevent slugs of water causing air locks in the tube. Fill the manometer tube until an equal level is visible about halfway up the scale, close valves (V6) and (V7).

Switch on reboiler heater power at the console and adjust power to the heater to 1.50 kW. The water in the reboiler will begin to heat up and this can be observed by selecting (T9) on the process temperature digital display.

As the water begins to boil, vapour will rise up the distillation column and this can be observed on the top plate of each of the two column sections as the insulation has been cut away in this area for this purpose. Turn the heater power down to 1.00 kW. Eventually vapour will reach the condenser and drops of condensate will be observed entering the glass decanter vessel (11). Allow water condensate to build up in the decanter vessel until it reaches the adjustable overflow and observe the condensate overflowing to the top product receiver vessel (10).

The reflux valve (12), when in the de-energised state (reflux control switch on the console is in the OFF position), will direct all condensate back to the top of the column as reflux.

Open valve (V10) at the base of the decanter and observe condensate flowing to the top of the column via the reflux valve. When a proportion of the condensate is required to be directed to the top product receiver vessel (10), it is necessary to set the reflux timer to give the ratio required. Set the timer to give 4 sec. to both column and receiver. This is achieved by first pressing the SET button on the reflux timer three times. The flashing digits are adjusted in turn using the (Δ) button. The CY- and CY+ digits in the bottom right of the display indicate flow of condensate back to the column and to the top product tank respectively.

Switch on the reflux control at the console. Observe condensate being directed to the column and receiver for the set time periods.

Observe the flows and temperatures for several minutes and allow the equipment to establish an equilibrium condition making slight adjustments where necessary. Ensure that all thermocouple sensors are giving sensible readings on the digital display at the console. In normal operation ensure that the power to the reboiler is not too high and the flow of cooling water is sufficient to prevent loss of vapour through the vent system (by overloading the condenser).

Open V6 and V7 and observing the pressure difference in the manometer. Close V6 and V7.

Open valve V14, allowing water to flow to the vacuum pump (20). Valve V14 MUST ONLY be operated with valve V5 OPEN. After a few minutes, observe on gauge P1 the pressure in the system begin to fall. Check for leaks. Even a very small leak of air into the system will affect the achievable vacuum. Allow the vacuum to reach a level of 200 mbar (-0.8 bar on P1). This should be achievable in 30-40 minutes from the time of opening V14. Close V14 and ensure that the non-return valve in the vacuum pump stops ingress of air and water. Break the vacuum by opening slowly, valve V15.

The equipment can now be shut down and allowed to cool. Switch off heater power and reflux valve at the console. Drain all of the water from the system using the drain valves V2, V3, V4 and V11.

It is important that all water be removed from the system as the presence of water with a mixture of solvents will adversely affect the experiments.

The UOP3CC may be used with a compatible PC and the supplied Armfield software, to provide data logging of sensor outputs and computer control of the apparatus.

The software is supplied on CD-ROM. To install the software use the following procedure:

- Insert the CD-ROM into the drive.
- The CD should autorun. If it doesn't, choose 'run' from the 'start' menu and type: *d:\setup.exe* where *d* is the letter of the drive you are using. Click 'OK'.
- Follow the instructions on the screen.

After restarting the PC, go to the PC 'start' menu, check to see that the UOP3CC software icon has appeared in the 'Armfield Process Control' group.

With the PC switched on, connect the USB port on the rear UOP3CC to a USB port on the computer, using the cable supplied. The red 'USB power' LED on the rear of the UOP3CC console should illuminate followed by the green 'USB active' LED. If the 'active' LED fails to light, disconnect the device, wait for about 30 seconds and then try again. If the LED still fails to light, re-start the computer and try again.

The PC will display a message saying that it has found new hardware. If the software is already installed then the new hardware wizard should find the files it requires automatically. This completes the software installation.

The basic operation of the UOP3CC has now been confirmed.

Refer to the Operation section in this manual for further information.

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