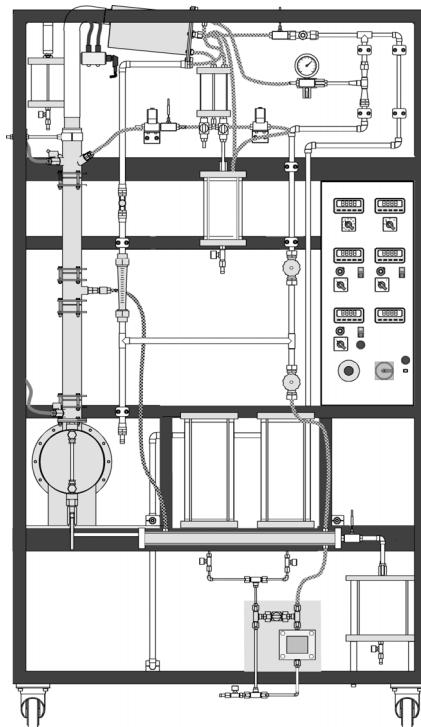


Experiment Instructions

CE 600 Continuous Rectification

CE 600**CONTINUOUS RECTIFICATION**

Experiment Instructions

Dipl.-Ing. (FH) Klaus Schröder

**Please read and follow the safety advices
before the first installation!**

10/2009



CE 600

CONTINUOUS RECTIFICATION

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1

Introduction

In physical-chemical engineering, liquid mixtures that consist of mutually soluble fluids are often separated by thermal processes. The main processes are **distillation** and **rectification**.

The **Continuous Rectification CE 600** experimentation unit enables liquid mixtures to be thermally separated by means of rectification in the experiment.

The unit consists of an electrically heated bottom in which the initial liquid mixture is heated and evaporated. The steam rises through a column and is subsequently condensed in a water-cooled condenser. From here, all of the condensate can be drained off as the top product, or all or part of it can be returned to the column.

Either a sieve plate column or a packed column can be used for the experiments. The column head incorporates a port for the return flow of distillate.

A pump can be used to pump mixture into the column, permitting continuous running of experiments. For that purpose, the sieve plate column features three ports at different heights. The separation behaviour varies depending on which port is chosen. The packed column features a port half way up.

A heat exchanger enables the pumped-in liquids to be pre-heated.

A glass filter pump can be used to generate a vacuum in the entire tube system for vacuum rectification.

The unit can be operated locally from the control cabinet. It is a good idea, however, to control the unit from a self-supplied PC using software supplied with the unit. This enables the available measurements to be automatically recorded and documented.

The range of experiments covers the following variants:

- Discontinuous (batch) or continuous
- Sieve plate column or packed column
- Sieve plate column, 3 different infeed heads selectable
- Reduction in the number of plates in the sieve plate column
- Operation at ambient pressure or under vacuum
- Operation with or without feed preheating

The instruction manual provides detailed instructions on performing the experiment. The method of operation is described in order to illustrate the possibilities and limits of the unit.

Potential series of experiments for the recommended ethanol/water material mix are demonstrated. Two experiments from the series are demonstrated, evaluated and commented upon in detail by way of example.

1.1 Proper use

The **Continuous Rectification experimentation unit CE 600** is intended solely for educational purposes and not for industrial production.

1.2 Teaching instructions

The unit is designed so that all operations are mostly visible and can be observed. The unit is for educational use in the field of thermal process engineering and apparatus construction.

A suitable laboratory facility is required to operate the unit (for water quality see chapter 5.6.1, page 47; for required cooling water flow see chapter 7.1, page 115). Control of the unit requires sound experience in the performance of experiments.

Areas of application of the unit are:

- **Demonstration experiments**

The tutor operates the prepared system while a small group of five to eight students observes. The reaction of the unit to changing conditions is demonstrated. The main effects can be demonstrated in one hour's operation.

- **Practical experiments**

Small groups of two to three students can also perform experiments independently. The estimated time required to perform an experiment is between four and five hours (from charging the unit and putting it into operation, recording data, determining the masses and concentrations through to draining the unit at the end).

- **Project work**

The unit is especially well suited to carrying out project work. The influence of changing conditions on the measurement results can be determined by way of experiment series.

In continuous mode, the experiments should be performed by two students. For batch experiments, a single, experienced student may also operate the unit.

Because of the complexity of the experimentation unit and the demanding subject, detailed training by a specialist GUNT engineer may be necessary and useful.

2 Unit Description

2.1 Process diagram

Fig. 2.1, page 6 shows the process diagram for the rectification unit CE 600.

This chapter sets out more information on the design of the unit and its components.

Information on the unit circuitry and instructions for performing the experiment in its different variants are provided in chapter 5.

CE 600

CONTINUOUS RECTIFICATION

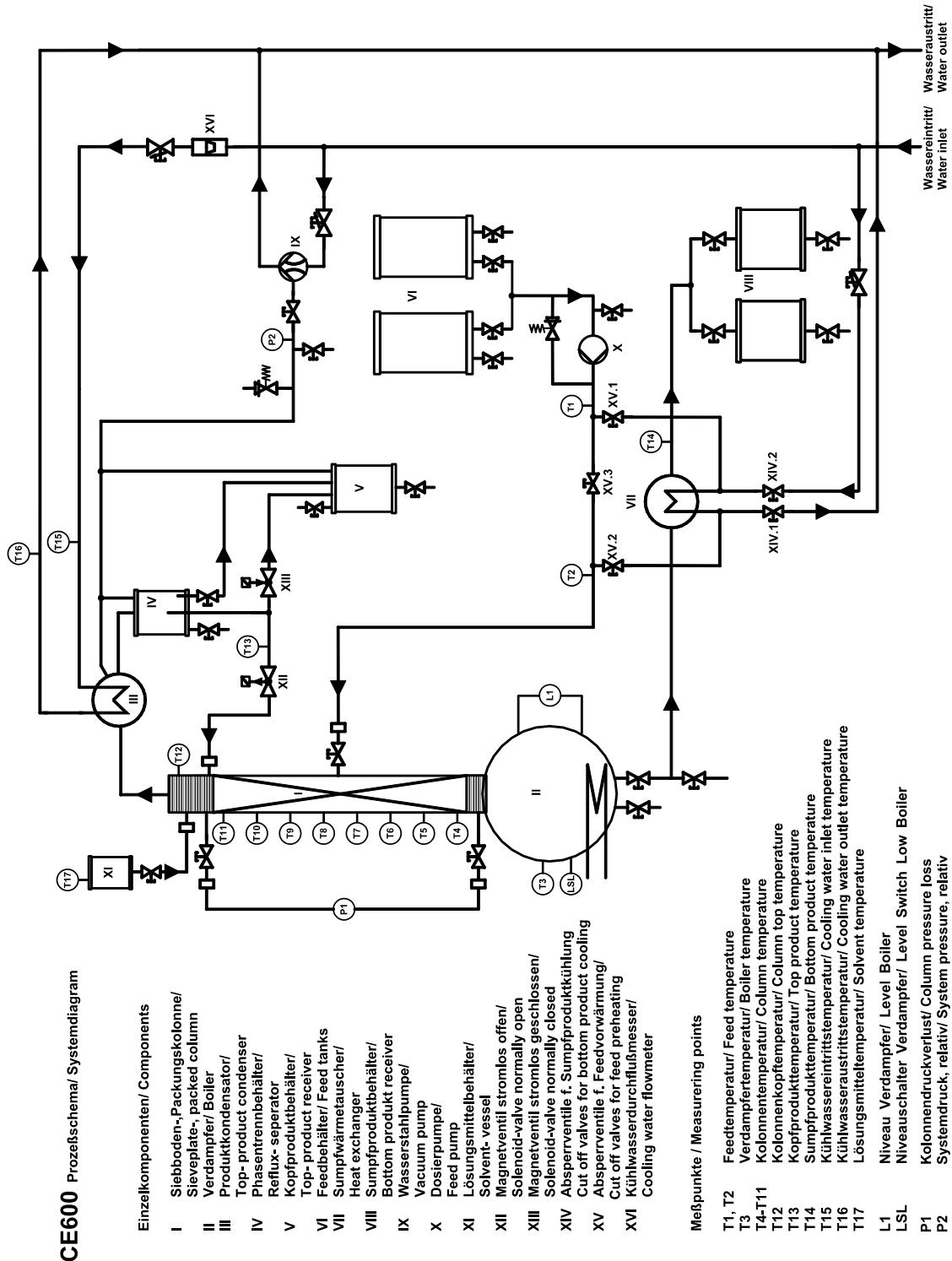
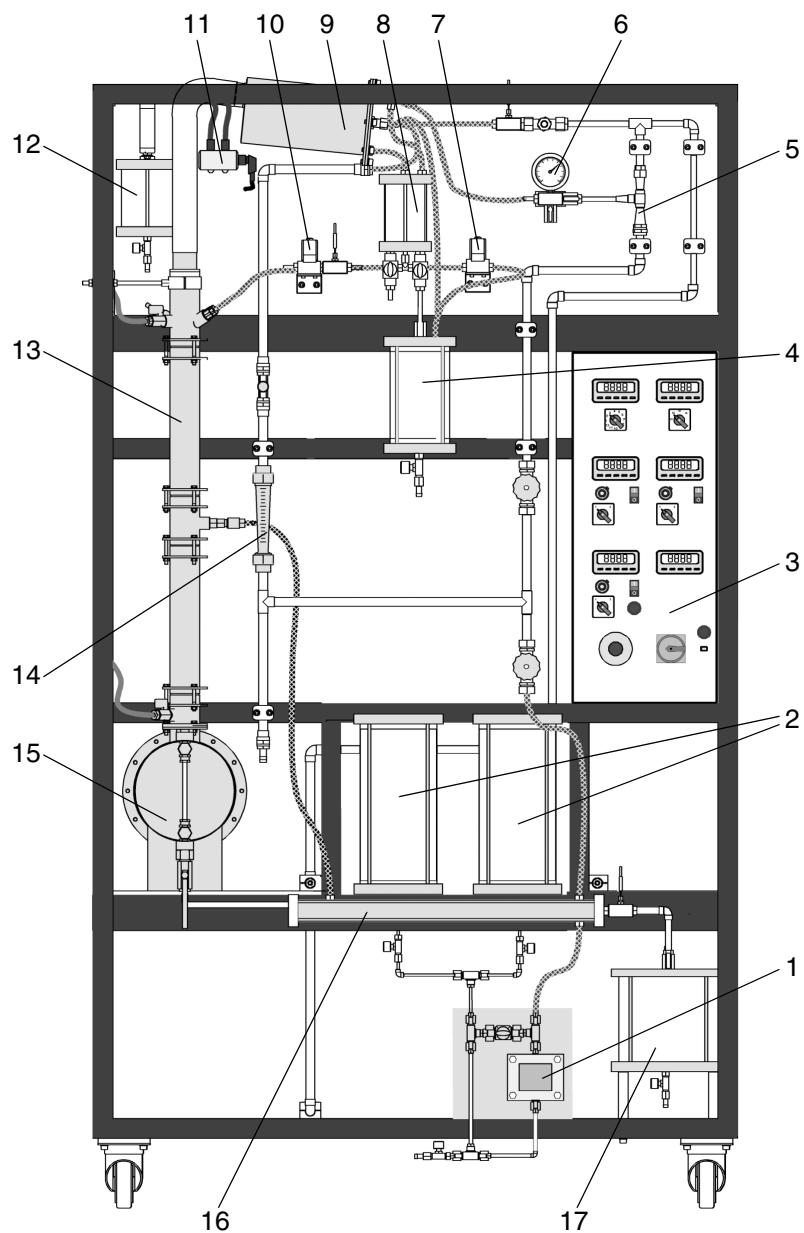


Fig. 2.1 Process diagram of rectification unit CE 600

CE 600**CONTINUOUS RECTIFICATION**

2.2 Layout

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Item	Name
1	Feed pump
2	Feed tank
3	Control cabinet
4	Top product tank
5	Glass filter pump
6	Pressure gauge
7	Switching valve
8	Phase separation tank
9	Product condenser
10	Switching valve
11	Differential pressure sensor
12	Solvent tank
13	Column
14	Flowmeter for cooling water
15	Evaporator
16	Bottom heat exchanger
17	Bottom product tank

Fig. 2.2 CE 600, Continuous Rectification, Overview

2.3 Equipment Functions and Components

2.3.1 Components for the thermal process

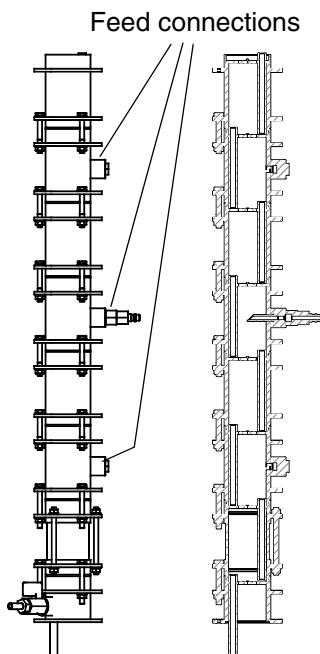


Fig. 2.3 Sieve plate column

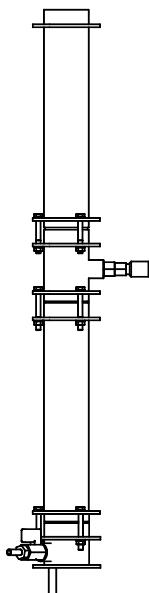


Fig. 2.4 Packed column

The **Continuous Rectification CE 600** experimentation unit is used to separate homogeneous liquid mixtures by means of rectification on an experimental scale.

A steel tank with a built-in electric heater acts as an **evaporator** (15) for the liquid mixture. The tank has level indicator tube on its front.

The evaporator is charged by means of the **feed pump** (1). The liquid mixture is pumped into the column from one of the two independent **feed tanks** (2).

One of the **columns** (13) in which the separation takes place is mounted on top of the evaporator. Either a packed column or a sieve plate column with eight plates can be used (see Fig. 2.3 and Fig. 2.4).

The pressure loss above the column is recorded by a **differential pressure sensor** (11).

The steam passes along a metal tube at the top of the column into the water-cooled **product condenser** (9). The distillate occurring at this point flows into a **phase separation tank** (8). From there, a tap enables it to be discharged directly into a **top product tank** (4). Using two electric **switching valves** (7, 10), the distillate downstream of the phase separation tank can also be split into reflux and top product with a defined reflux ratio. The reflux returns to the unit by way of the column head.

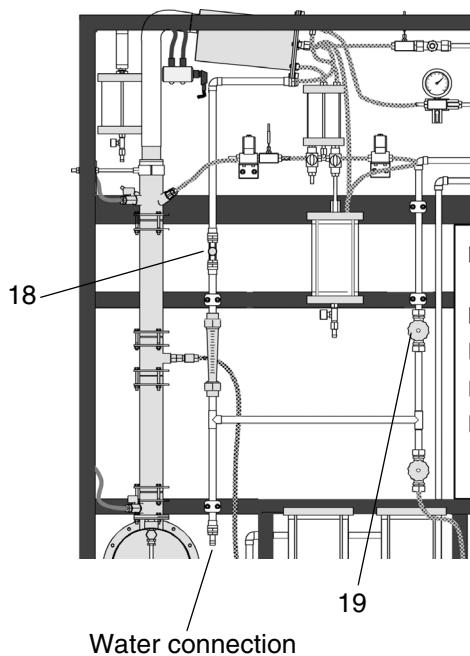


Fig. 2.5 Cooling water connection

The **cooling water** for the product condenser must be supplied from an external source via a laboratory connection (for position of connection see Fig. 2.5). The cooling water volumetric flow can be read from the **flowmeter** (14). This volumetric flow is regulated by way of the hand valve (18) above the flowmeter (see Fig. 2.5).

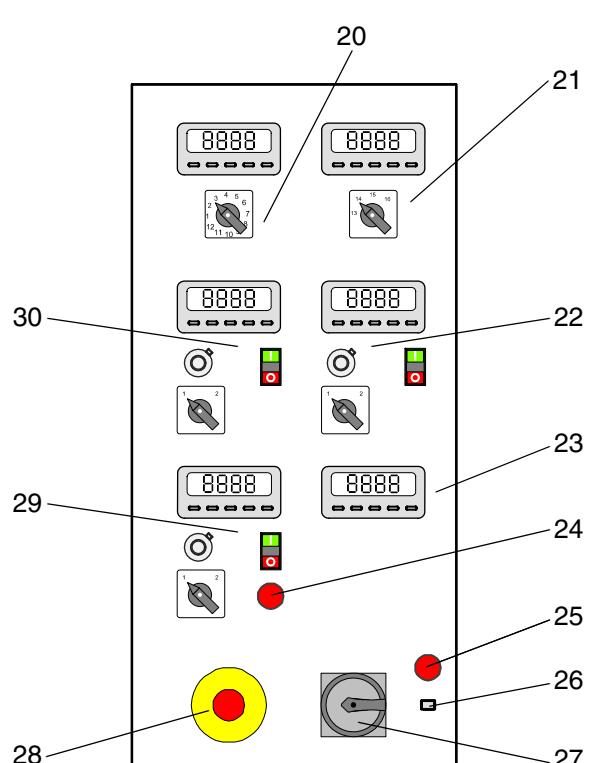
The residue which is enriched in the evaporator during the experiments is called the bottom product. The bottom product can be drained directly into a suitable vessel by way of a tap on the front of the evaporator. Alternatively, it can be discharged into one of the **bottom product tanks** (17). In doing so, it passes through the **bottom heat exchanger** (16). This can be used to preheat the **feed** (mixture infeed) with the waste heat of the draining bottom product when the unit is operating in continuous mode. If this feed preheating is not required, the bottom heat exchanger can be operated with cooling water from the laboratory connection to cool the bottom product.

A **glass filter pump** (5) is used to perform experiments with vacuum. The pump reduces the pressure throughout the system. The pump is also operated with water from the laboratory supply. The hand valve (item 19 in Fig. 2.5) is used to regulate the flow of water through the pump. The level of vacuum can be read from the **pressure gauge** (6).

2.3.2 Control cabinet

Some switching and monitoring functions can be provided by the electrical **control cabinet** (3).

All available data can be measured and recorded, and all control functions can be implemented, by means of the separate program on PC.
(see chapter 2.4.2, page 13).



Item	Name
20	Temperature display T1-T12 with measuring point selector switch
21	Temperature display T13-T16 with measuring point selector switch
22	Controls for the reflux ratio
23	Display for the differential pressure of the column
24	"Low Level" warning lamp (lack of liquid in the evaporator)
25	"Watchdog" warning lamp
26	USB port
27	Master switch
28	Emergency Stop button
29	Controls for the feed pump
30	Controls for the heater

Fig. 2.6 Control cabinet, front view

The following unit components can be operated from the control cabinet:

- Heater (30)
- Feed pump (29)
- Reflux ratio (22)

The controls listed below are provided for the purpose:

- I/O switch
- Adjusting knob
- Local/Auto selector switch, with:
Local: Settings made on control cabinet
Auto: Control from PC

Digital displays are installed for these unit components and for the differential pressure and the temperatures.

Additional control cabinet functions:

- “Watchdog” warning lamp (25), signals failure of the external controller (PC)
- “Low Level” warning lamp (24)
The evaporator is fitted with a **level switch**. To prevent overheating, if the level falls below the **minimum** (~3L), the heater is automatically deactivated. The warning lamp is also activated.
- On the rear of the control cabinet there are sockets to connect the temperature sensors T4-T11 of the column (see Fig. 2.7).

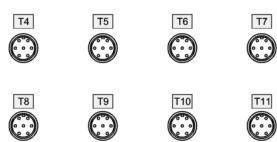


Fig. 2.7 Control cabinet, rear panel

2.4 Data acquisition program

2.4.1 Installing the program

The following is needed for the installation:

- An fully operational PC, laptop or notebook with USB port (see Appendix for minimum requirements).
- G.U.N.T. - CD-ROM



Note! All components necessary to install and run the program are contained on the CD-ROM, delivered by G.U.N.T. along with the CE 600. No further aids are necessary!

After starting, the installation runs automatically. During the course of the installation, various program components are loaded onto the PC

- LabVIEW®- runtime program for PC data acquisition
- Driver routines for the “LabJack®” USB converter

Installation procedure



Note! Connect nothing to the USB port during the installation of the program. Only after the software has been installed can the USB hardware be connected.

- Boot the PC
- Load the G.U.N.T. - CE 600 CD-ROM
- Start the installation program “**Setup.exe**” in the folder “**Installer**”.

- Follow the installation procedure on the screen.
- Reboot the PC after the installation is finished.

2.4.2 Operating the program

The program for the CE 600 is selected and started by choosing:

Start / All Programs / G.U.N.T. / CE600

When the software is run for the first time after installation, the language to be used for the program is requested (once only).

Note! The language selected can subsequently be changed at any time on the “**Language**” menu.

The Continuous Rectification CE 600 system diagram then appears on-screen (see Fig. 2.9).

It shows the available system data along with the associated measuring point positions.

The following elements can also be controlled from the PC:

- Heater (T3)
- Reflux ratio (0...100%)
- Feed pump (0...100%)



Fig. 2.8 Language selection

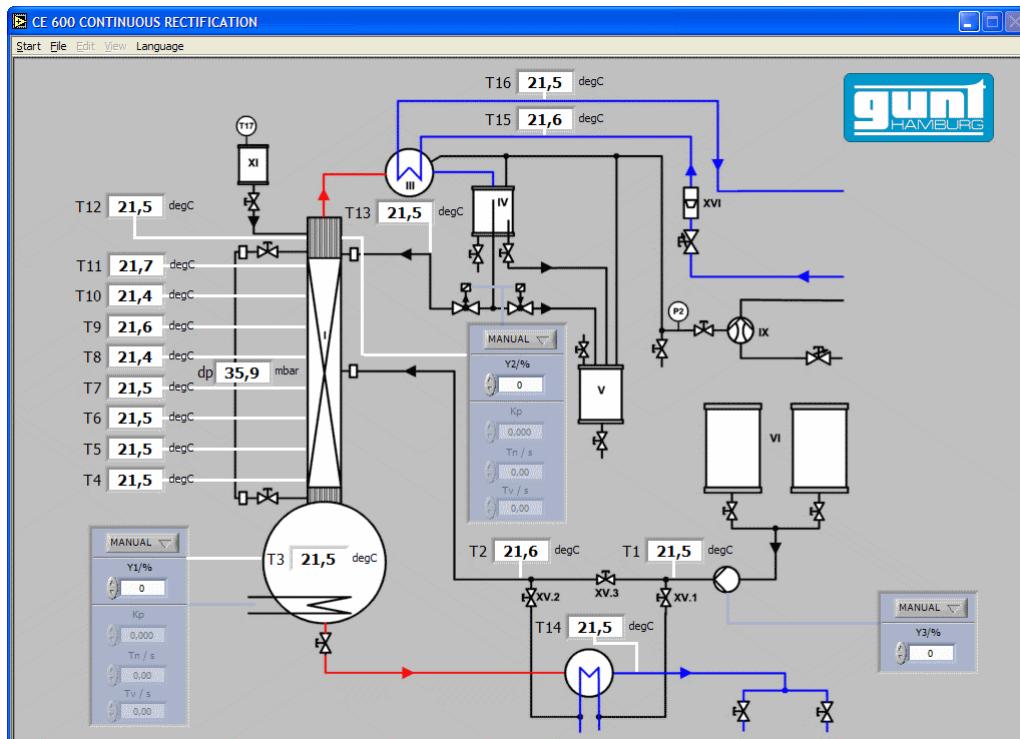


Fig. 2.9 Process diagram, System diagram

One of two modes can be selected:

- **Manual:**
The desired value is directly entered (0...100%)
- **Auto:**
The PC functions as a controller. Setpoint values and control parameters are entered on the PC.

Note regarding Auto mode:

The actual set of control parameters will be stored. If required, the default set of control parameters can be activated again (therefor click „File“ and „load default values“).



The feed pump cannot be controlled automatically. It features only the **Manual** mode.



To access a display of the progression of the signals over time, use the "Start/Charts" pull-down menu. In this dialog box the measured signals are displayed as curves.

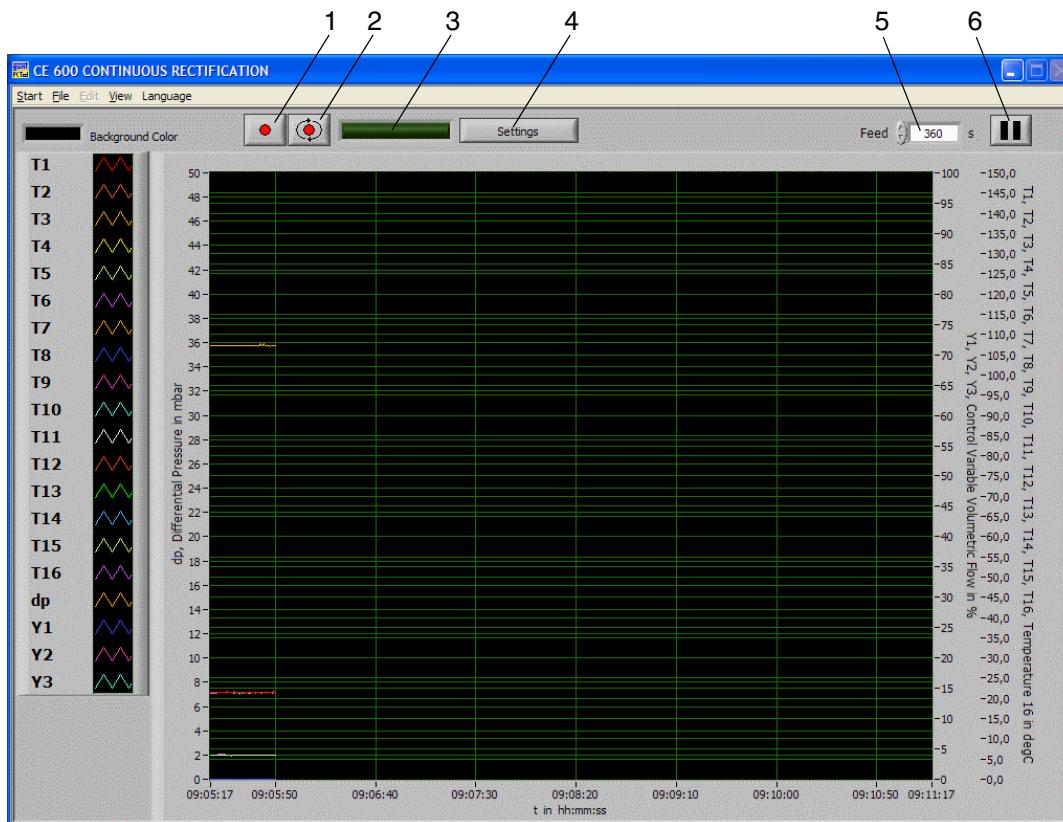


Fig. 2.10 "Charts/Time progression" dialog box

The axes of the curve view can be scaled. To do this, click on the start or end value and type in the new value using the numeric keypad. Click the mouse or press "Enter" to confirm.

Additional functions:

- 5: Enter the time span for the graph.
- 6: Start/stop the chronological advance of the curve.

Measured value files can also be defined and recorded in this dialog box.

A **measured value file** can consist of more than one **series of measurements**.

Each series is divided into a **header** and the **measured data field**. The measured data field contains a chronological sequence of measured data records. A **measured data record** contains the current measured value for each individual measuring point.

- 1: Write the current measured data record to the measured value file once ("single shot").
- 2: Click this button to start automatic recording, according to the setting in the dialog box detailed below (see Fig. 2.11).
- 3: The green field briefly lights up to indicate each time that measured data records are written to the file.
- 4: Click the "Settings" button to open another dialog box:

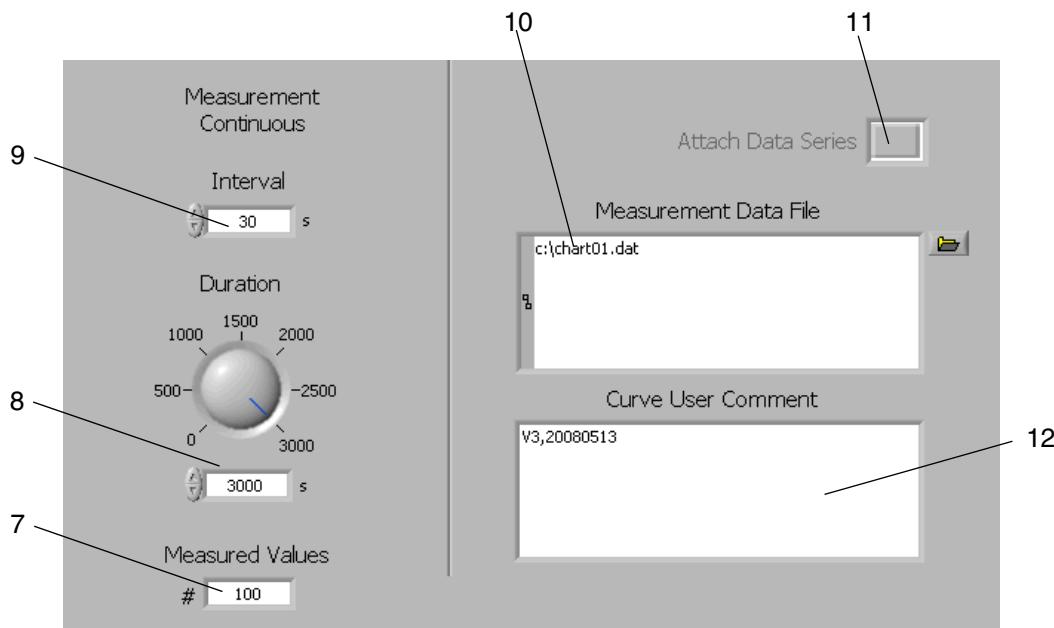


Fig. 2.11 "Settings" dialog box

- 7:** The number of measured data records to be stored is entered here.
- 8:** The duration of continuous measurement is defined here, either by turning the pointer or by typing the figure in the white field.
- 9:** The interval for automatic recording of measured data records is defined here (time between recording of two measured data records).
- 10:** A name and a storage location for the measured value file to be created must be entered.
- 11:** This box may be ticked or unticked. If it is unticked, the new measured data records are attached to the previous ones in the same measured data field. If the box is ticked, a new series of measurements is created in the existing measured value file.
- 12:** This box can be used to enter any comments, such as the experiment number, details of background conditions and so on. The comments are displayed in the measurement series header.

2.5 Commissioning

- Ensure that all hose connections are secure and free from leaks
- Connect the cooling water supply and reflux lines (see Fig. 2.5, page 9), reflux unpressurized
- Connect the unit to the mains electricity supply
- Install the data acquisition and control program on the PC (see chapter 2.4.1, page 12)
- Plug the unit into the PC

For more information and detailed instructions for performing the experiment refer to chapter 5 and chapter 6.

2.6 Shutdown

- Shut down the PC
- Switch off the unit at the master switch
- Shut off the water supply and isolate the unit from the water main
- Leave unit to cool
- Check the positions of the hand valves, prevent unintentional leakage of liquids
- If the unit is being shut down for a lengthy period of time, drain all tanks completely

3**Safety**

The experiment instructions, in particular the safety instructions, must be read thoroughly prior to starting up the unit. Before commencing an experiment, all participants must have been instructed in the safety aspects and proper handling of the unit.

3.1**Health hazards****⚠ WARNING****Risk of electric shock when working on the control cabinet**

Have work on the control cabinet carried out only by a qualified electrician. Prior to opening the control cabinet, unplug the mains power plug.

⚠ WARNING**Risk of electric shock due to wetness and moisture on the control cabinet**

Do not allow the control cabinet to get wet.

**⚠ WARNING****There is a risk of explosion if the wrong chemicals are selected.**

Select chemicals such that ignition temperatures cannot be reached during operation. This applies to the single input materials and to mixtures of them.



**⚠ WARNING**

Potential health hazard when handling chemicals.

Follow the relevant health and safety instructions, particularly with regard to charging and draining the unit.

**⚠ WARNING**

Risk of burns by touching the apparatus, in particular the evaporator, the column and the pipes.

Do not touch hot surfaces.

**⚠ WARNING**

Risk of scalding when removing the heated bottom product.

Drain the heated bottom product only into a heat-proof container, and wear heat-resistant gloves while doing so.

3.2 Hazards to the unit and its function

**NOTICE**

The choice of chemicals for rectification is limited by the resistance of the materials used in the construction of the equipment. Use only chemicals which do not attack VITON, PVDF, PTFE and stainless steel!

4 Theory

4.1 Basic principles

Distillation and rectification are thermal processes used to separate or purify liquid mixtures whose constituents are wholly miscible with one another.

Both processes work by the basic operations of evaporating a liquid and then condensing the resulting vapours.

When a liquid mixture **AB** is brought to the boil, wherein the substance **A** has the **molar fraction** x_1 , vapour arises from it. But substance A now has a different molar fraction y_1 in the steam than it does in the liquid mixture AB.

If we enter the corresponding values for the vapour for all possible mixture ratios of AB in a diagram, we obtain the **curve of equilibrium** (see Fig. 4.1). This curve indicates the mixture ratio of the steam given off by the boiling liquid.

Curves of equilibrium are different for different substance mixtures. The more the curve arcs away from the diagonal of the diagram, the greater is the increase in concentration of substance A from liquid to vapour.

The collected condensate is therefore richer in low-boiling constituents compared with the initial mixture. The proportion of high-boiling constituents in the residual mixture is correspondingly higher.

In distillation/rectification therefore, the vapour phase has a different mixture ratio than the liquid phase. This fact is the basis of both processes.

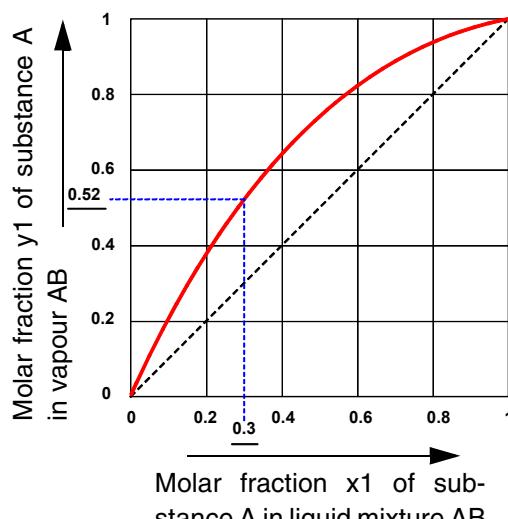


Fig. 4.1 Example of a curve of equilibrium

4.1.1 Distillation

In distillation, a liquid mixture is brought to the boil and the resulting vapour is drawn off and condensed. This condensed liquid is known as the **distillate**. The remaining mixture is the **residue**.

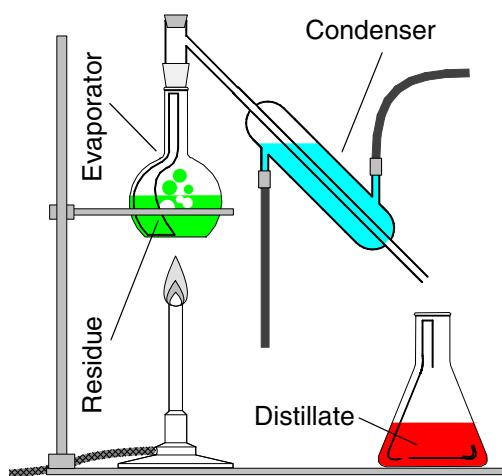


Fig. 4.2 Distillation, schematic

Distillation apparatus consists of:

- an **evaporator** to heat the initial mixture and collect the high-boiling component (residue).
- a **condenser** to condense the low-boiling component (distillate).
- one or more tanks to collect the distillate.

Distillation does not produce a complete separation of the initial mixture. It just divides the initial mixture into two mixtures with different concentrations (see chapter 4.1, page 21).

In **simple distillation**, all of the distillate is collected.

There is also the process in which the condensate is collected in different vessels, known as **fractions**, as distillation time passes. This is known as **fractional distillation**.

To protect temperature-sensitive substances during distillation, their mixtures are distilled in a vacuum where boiling points are lower. This method is known as **vacuum distillation**.

This process is essentially just the same as conventional distillation. A vacuum pump downstream of the condenser maintains a vacuum throughout the system.

There are two basic types of distillation:

- Batch distillation (discontinuous, batch-by-batch):
The evaporator is charged and the distillation is continued until no more distillate occurs for example.
- Continuous distillation:
Distillate and residue are continuously discharged. To compensate for the discharge, a corresponding amount of additional initial mixture (feed) is pumped into the evaporator.

4.1.2 Rectification

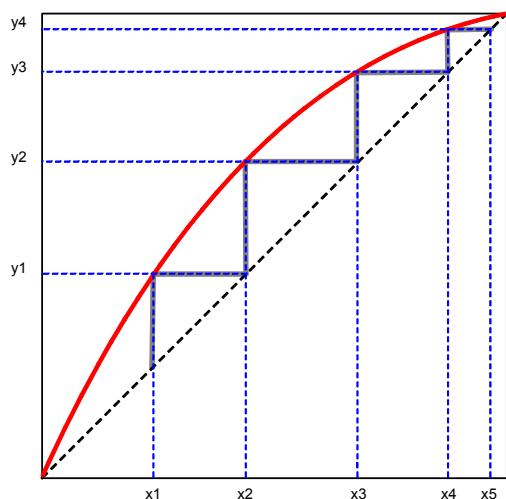


Fig. 4.3 Equilibrium diagram for multi-stage distillation

The result of the separation obtained by distillation is not sufficient in all cases.

To improve separation, the collected distillate should be distilled again. This process should be repeated until the required material is satisfactorily enriched. **Multiple-stage distillation** of this type is expensive and energy-intensive.

Consequently, **rectification** is applied (for a schematic view see Fig. 4.4 and Fig. 4.5).

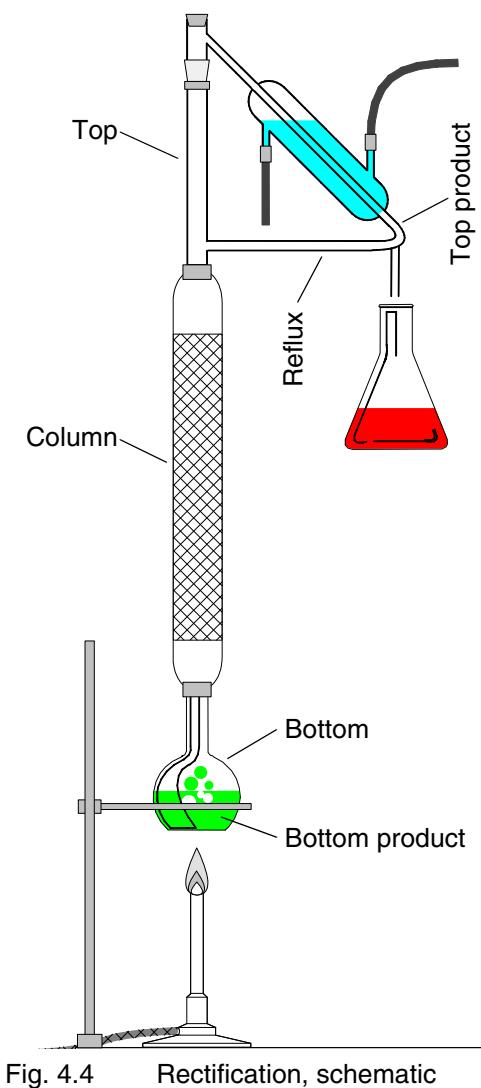
In rectification, the released vapour is first passed through a vertical tube called the **column** before it reaches the condenser. Instead of all of the distillate collected in the condenser being drawn off straight away, part of it is returned to the column as the so-called **reflux**.

The difference between rectification and distillation is that the rising vapour mixture and returning condensate flow counter to one another. The process is therefore also known as **reverse flow distillation**.

The returned condensate now runs down the column and, at the next level down, is forced into contact with the rising vapours on suitable internals for the purposes of material exchange.

The result is an exchange of material and heat between the rising vapours and the falling liquid.

The high-boiling component condenses first from the rising vapours.



The resulting condensation heat causes more low-boiling component to evaporate in turn. The result is that the liquid flowing back to the **bottom** of the column is enriched with high-boiling components. The vapours at the **top** of the column are correspondingly richer in low-boiling components.

Rectification can be understood as being **multi-stage distillation**, but with just one evaporator and one condenser. This improves efficiency, because at the transition from one **stage** to the next the vapour does not have to be first condensed and then reheated and evaporated.

Thus the number of stages in rectification is comparable to the number of series-connected distillation stages.

If the column is a **sieve plate column** (for illustration see Fig. 2.3, page 8 and Fig. 5.8, page 35), each sieve plate represents one separation stage. The number of sieve plates in the column is the **plate count**.

A common alternative to the sieve plate column is the **packed column**. It is filled with packing. The purpose of the large number of packing blocks is to provide the material mixture with a large surface area. This intensifies the material exchange in the column. The contact between the liquid and gaseous phases occurs throughout the packed bed.

Packing blocks are small, regularly shaped pieces of plastic, ceramics or metal.

There is a wide variety of different packing blocks to suit different separation applications.

The packed column, with its homogeneous packed bed, has no direct plate count. To be able to characterise and design packed columns despite this, the concept of **theoretical transfer units** has been devised. In this, **NTU** (Number of Transfer Units) represents a theoretical number of transfer units and **HTU** (Height of Transfer Unit) is the height of the packed bed of such a unit.

At the top of the column, the distillate is divided into one part reflux and one part top product. The ratio of reflux to top product is called the **reflux ratio**. It is an important characteristic value for operation of the unit. The reflux ratio v may assume values from zero to infinity.

As already described in chapter 2.3.1, page 8, the CE 600 unit separates distillate into reflux and top product by cycling two electric switching valves. In this process, the switching valves 7 and 10 are alternately opened and closed.

Differing from the usual definition, an adjusted definition is used as the **reflux ratio R** for this unit. Here R is the ratio of the opening time of switching valve 10 to the sum total of the opening times of switching valves 7 and 10. The value range for R is 0 to 100%.

The following examples illustrate this reflux ratio R:

- $R=100\%$: Switching valve 10 is permanently open, switching valve 7 permanently closed. The entire condensate flows back into the column.
- $R=0\%$: Switching valve 10 is permanently closed, switching valve 7 permanently open. The entire condensate flows into the top product tank.
- $R=50\%$: Switching valves 10 and 7 are alternately opened and closed, and for the same amount of time. The condensate flow is divided. One portion flows back into the column, the other is routed to the top product tank.

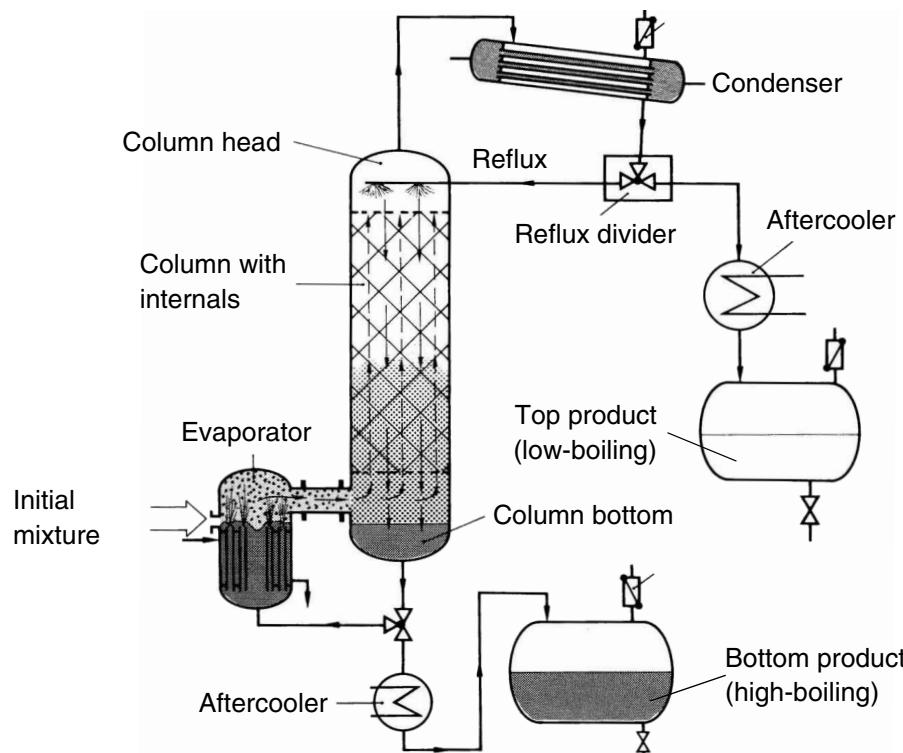


Fig. 4.5 Rectification unit, schematic representation

Rectification can also be carried out in two different ways:

- Batch rectification (discontinuous, batch-by-batch):
The evaporator is charged and the rectification is continued until no more top product occurs for example.
- Continuous rectification:
The top and bottom products are continuously discharged. To compensate for the discharge, a corresponding amount of additional initial mixture (feed) is pumped through the column.

Liquid mixtures can be separated virtually completely by rectification. Even mixtures with curves of equilibrium close to the diagram diagonal are separated much more effectively than in single-stage distillation.

4.1.3 Azeotropic mixtures

A special case occurs with regard to many liquid mixtures when they reach a certain concentration. The vapour arising from the mixture has the same composition as the liquid phase. Such mixtures are called **azeotropic**.

This means that the curve of equilibrium intersects the diagram diagonal.

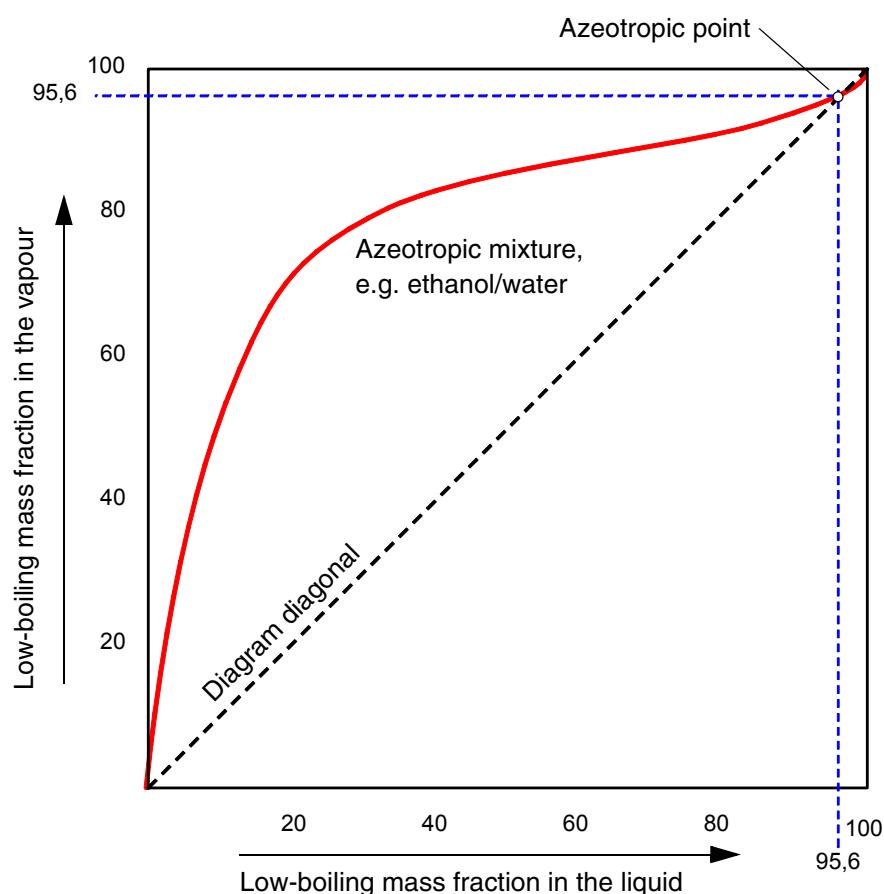


Fig. 4.6 Curve of equilibrium of an azeotropic mixture

Normal rectification can only separate such mixtures down to their azeotropic point.

The closer the separation gets to that point, the more similar the compositions of the liquid and vapour become. Consequently, no further enrichment of the lower-boiling component in the condensate is possible. For an ethanol/water mixture for example, this concentration is 95,6%*m* (mass fraction).

Two methods can be applied to obtain a pure component:

- Altering the working pressure in the column by vacuum or high-pressure distillation. This enables the azeotropic point to be shifted or eliminated in many cases.
- Addition of an ancillary substance to the mixture. This substance forms a low-boiling or high-boiling mixture together with one of the components. This likewise enables the azeotropic point to be shifted.

This process is termed **azeotropic rectification**. For an ethanol/water mixture for example, cyclohexane is a suitable ancillary substance. It is intermingled with the product containing approximately 95%*m* ethanol. The product is separated into the now higher-boiling ethanol, which is enriched in the bottom of the column, and the now lower-boiling water/cyclohexane mixture.

Water and cyclohexane are not miscible. Owing to their different densities, they are separated in the phase separation tank. The lighter cyclohexane floats to the top and is fed back into the column. This means the ancillary cyclohexane circulates around the system.

5**Experiments - Preparations, variants, instructions for performing**

The following provides instructions for carrying out rectification with the CE 600 unit.

This section occasionally refers to CE 600 components without showing the process diagram adjacent to the text. For clarification, please refer to the process diagram in Fig. 2.1, page 6.

5.1**Preparing the unit**

First run through the commissioning procedure as detailed in chapter 2.5, page 18.

5.1.1**Tank volumes**

When performing the experiments, it is useful to be able to obtain a quick estimate of the levels in the various tanks.

This is why the following tanks have level markers (Fig. 5.1 to Fig. 5.4, page 32):

- feed tank
- evaporator
- top product tank
- bottom product tank

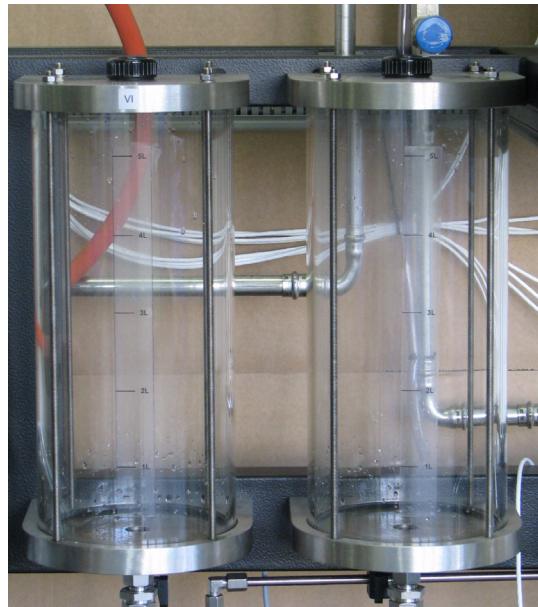
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Fig. 5.1 Feed tank (VI) volume



Fig. 5.2 Top product tank (V) volume

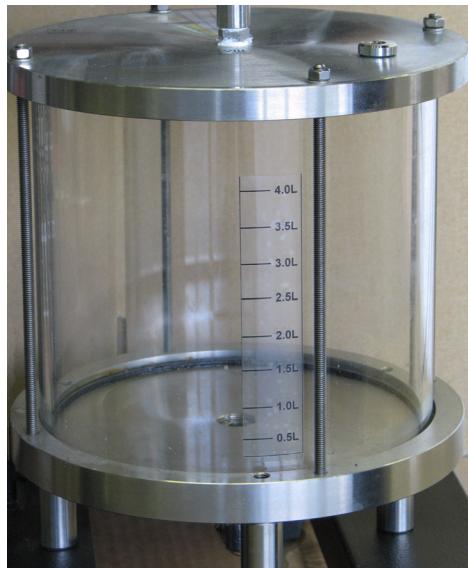


Fig. 5.3 Bottom product tank (VIII) volume

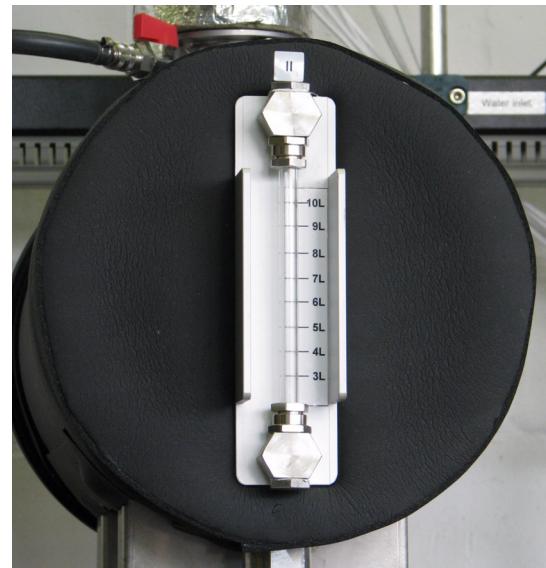
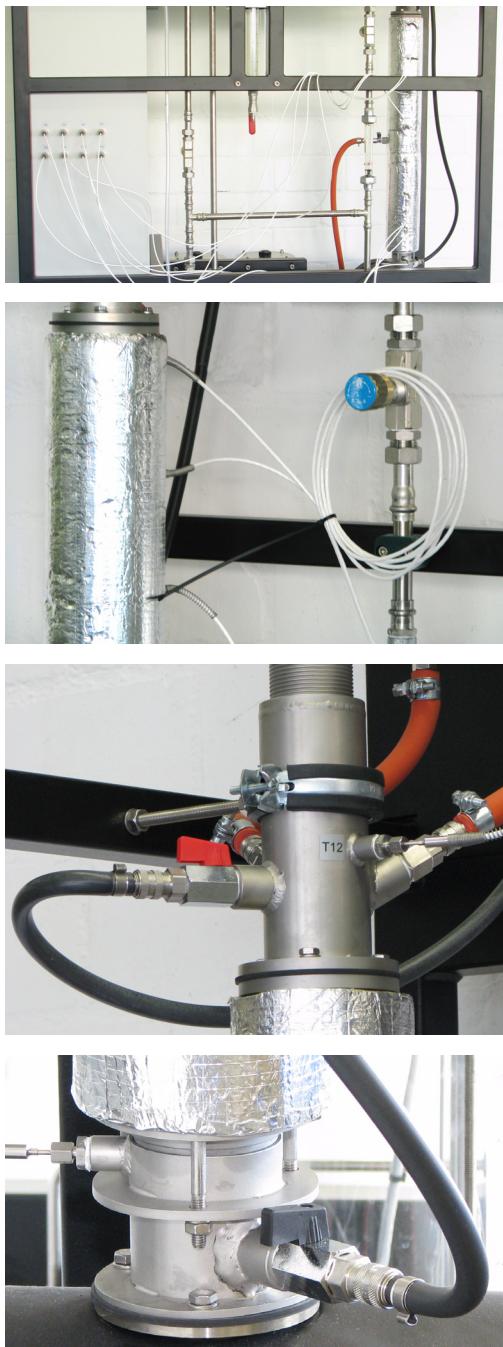


Fig. 5.4 Evaporator (II) volume

5.1.2 Refitting the columns

5.1.2.1 Replacing the columns



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To convert between a sieve plate and packed column, run through the following steps (see adjacent photos in Fig. 5.5).

- Make sure the unit has cooled down sufficiently.
- On the rear panel of the control cabinet, detach and unplug the connectors of the measuring cables for temperature sensors T4 to T11.
- Reel up and tie the cables in pairs, using a cable tie for example.
- Detach the hoses from the column hose couplings.
- Release the clamp around the column head.
- Unscrew and remove the screws on the top and bottom column flanges.
- With the aid of a second person, push the column head up slightly and remove the column. Store the column carefully, so that the temperature sensors are not bent.
- Clean the two flange seals (see Fig. 5.6) and coat them on both sides with water (for explanatory notes see below).
- With the aid of a second person, carefully fit the replacement column with the flange seals.
- Fit the screws, retaining rings and nuts on the top and bottom column flanges and tighten them evenly.

Fig. 5.5 Column photos



Fig. 5.6 Flange seal

- Clamp around column head.
- Unreel the tied-up measuring cables.
- On the rear panel of the control cabinet, insert and secure the connectors of the measuring cables for temperature sensors T4 to T11. Keep to the assignments labelled on the connectors and on the rear panel socket.
- Connect the hoses to the column by their couplings.

The flange seals tend to adhere more strongly to the flanges as the service life advances. This makes removing them more difficult. The adhesion can be reduced by coating the flange seals with water prior to fitting.

5.1.2.2 Sieve plate column, plate removal



Fig. 5.7 Sieve plate column mounting

For the sieve plate column, it is useful to investigate the influence of the number of plates. The following section explains how individual plates can be removed from the column and re-inserted into it.

Fig. 5.7 shows how the disassembled sieve plate column can be clamped to a bench.

The following photos in Fig. 5.8 and Fig. 5.9 show the actions required. The following steps must be run through to remove a sieve plate:

- Clamp the sieve plate column to the bench
- Slacken and remove the flange screws at the desired separation point

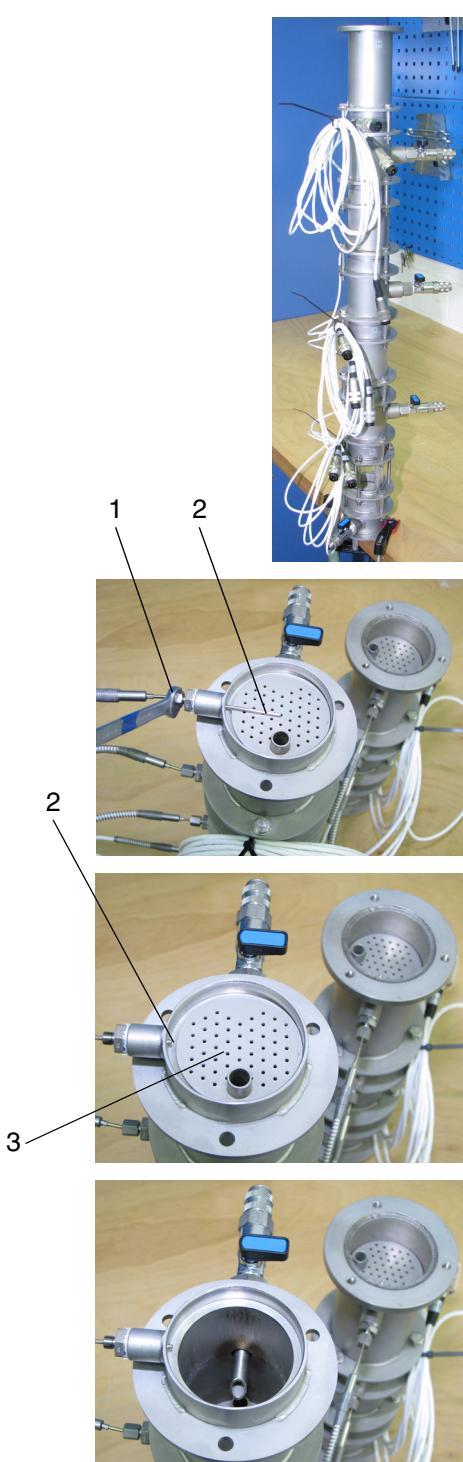


Fig. 5.8 Photos: Disassembling a sieve plate column

- Detach the upper section of the column
- Release the temperature sensor mount (1)
- Pull back the temperature sensor (2)
- Remove the sieve plate (3)
- Assemble the distance washer (see Fig. 5.9, item 4), replacing the removed sieve plate (3)
- Return the temperature sensor (2) to its original position, and tighten the union nut
- Moisten the O-ring (see Fig. 5.9, item 5) and ensure it is firmly seated
- Carefully slot the upper section of the column at the correct angle (not twisted) into the bottom section (without tilting it).
- Fit the screws, retaining rings and nuts. Tighten the nuts carefully and evenly.

Purpose of the distance washer: Five distance washers are part of the accessories. The assembled distance washer replaces the removed sieve plate, in order to get the identical overall height of the column, and to avoid damages of the temperature sensor (without distance washer, there is a risk that the temperature sensor between the upper and lower sections of the column may be crashed).

To add a single sieve plate, follow the above procedure in reverse.



Fig. 5.9 Photos: Assembling a sieve plate column

The photos illustrate that the space between the flanges is required for removal and fitting of the sieve plates. Consequently, there is no room for insulation on a sieve plate column. By contrast, a packed column is supplied with insulation, so as to reduce heat loss.

5.2 Accessories for performing experiments

To perform the experiments, a number of accessories are required in addition to the actual unit and the material mixture under investigation (accessories not included in the scope of delivery).

5.2.1 Tanks, measuring cups

- Three measuring cups, e.g. 2000ml capacity
- Two lockable canisters, e.g. 25L capacity, for interim storage of the substances and mixing
- A shallow cup, e.g. 500ml, for removing bottom product from the bottom product tanks.

5.2.2 Measuring instruments

- A weighing scale, measuring range up to at least 2,5kg
- A concentration meter for the material mixture (e.g. density spindle/areometer, with upright cylinder)
- A thermometer to measure the sample temperature

5.3 Experiment variants

The unit offers a wide range of options for carrying out experiments under widely varying conditions.

The possible modes include:

- Discontinuous (batch) or continuous
- Sieve plate column or packed column
- Sieve plate column, three different infeed heads selectable
- Reduction in the number of plates in the sieve plate column
- Operation at ambient pressure or under vacuum
- Operation with or without feed preheating
- Operation of heater and reflux ratio via fixed values, or regulated

The key difference in terms of operating mode is between discontinuous (batch) and continuous. This differentiation also determines the basic method of performing the experiment.

5.4 Experiments - batch mode

The batch experiments differ primarily in that the unit is operated at ambient pressure or under vacuum. The differences with regard to performing experiments are set out in the following subsections.

5.4.1 Notes on performing experiments - operation at ambient pressure

Fig. 5.10, page 40 is a variant of the actual process diagram in Fig. 2.1, page 6, and the flow through the unit for batch experiments at ambient pressure in colour-coded form.

The unit components, pipe lines and hoses are colour-coded. The meanings of the various colours are indicated by the key.

Before performing an experiment, the manual stop valves must be set to produce the flow shown during subsequent operation.

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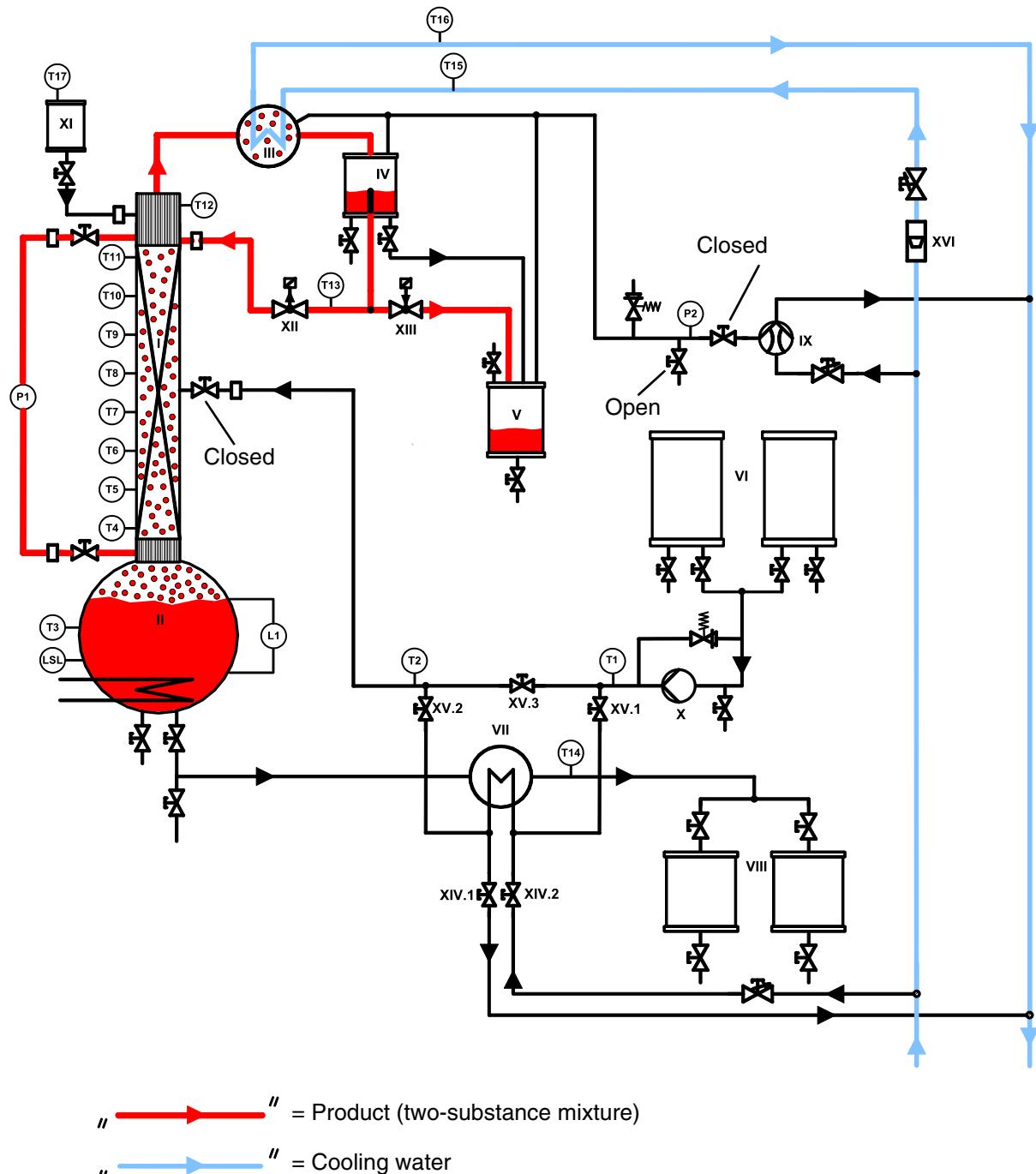


Fig. 5.10 Process diagram for a batch experiment at ambient pressure

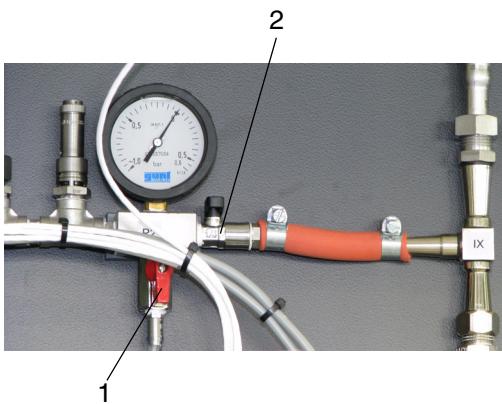


Fig. 5.11 Ambient pressure circuit

The process diagram Fig. 5.10 also specifies the positions of the two small stop valves close to the pressure gauge. They are shown in the adjacent photo Fig. 5.11 (items 1 and 2).

For operation at ambient pressure it is important to open the manual stop valve (1) underneath the pressure gauge. This equalises the pressure relative to the atmosphere.

If the unit was operated with the stop valve (1) closed, the entire system may be pressurised because of the rising steam pressure. The consequence of the higher pressure would then be a higher boiling point, meaning that the temperature level throughout the system would also rise. Such an increased temperature level may result in the destruction of unit components.

For the same reason, it is advisable to open the stop valve in order to vent the top product tank (V).

The stop valve (3) shown in Fig. 5.12 at the feed connection of the column should be closed so as to avoid unnecessary thermal loading on the hose.

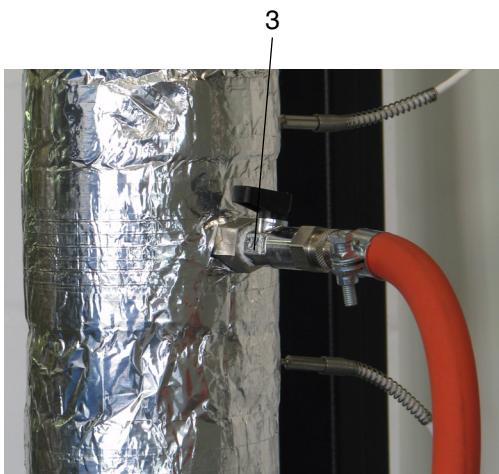


Fig. 5.12 Column, feed connection

5.4.2 Notes on performing experiments - operation with vacuum

Fig. 5.13, page 42 is also a variant of the actual process diagram in Fig. 2.1, page 6, and represents the flow through the unit for batch experiments under vacuum in colour-coded form.

Before performing an experiment, the manual stop valves must be set to produce the flow shown during subsequent operation.

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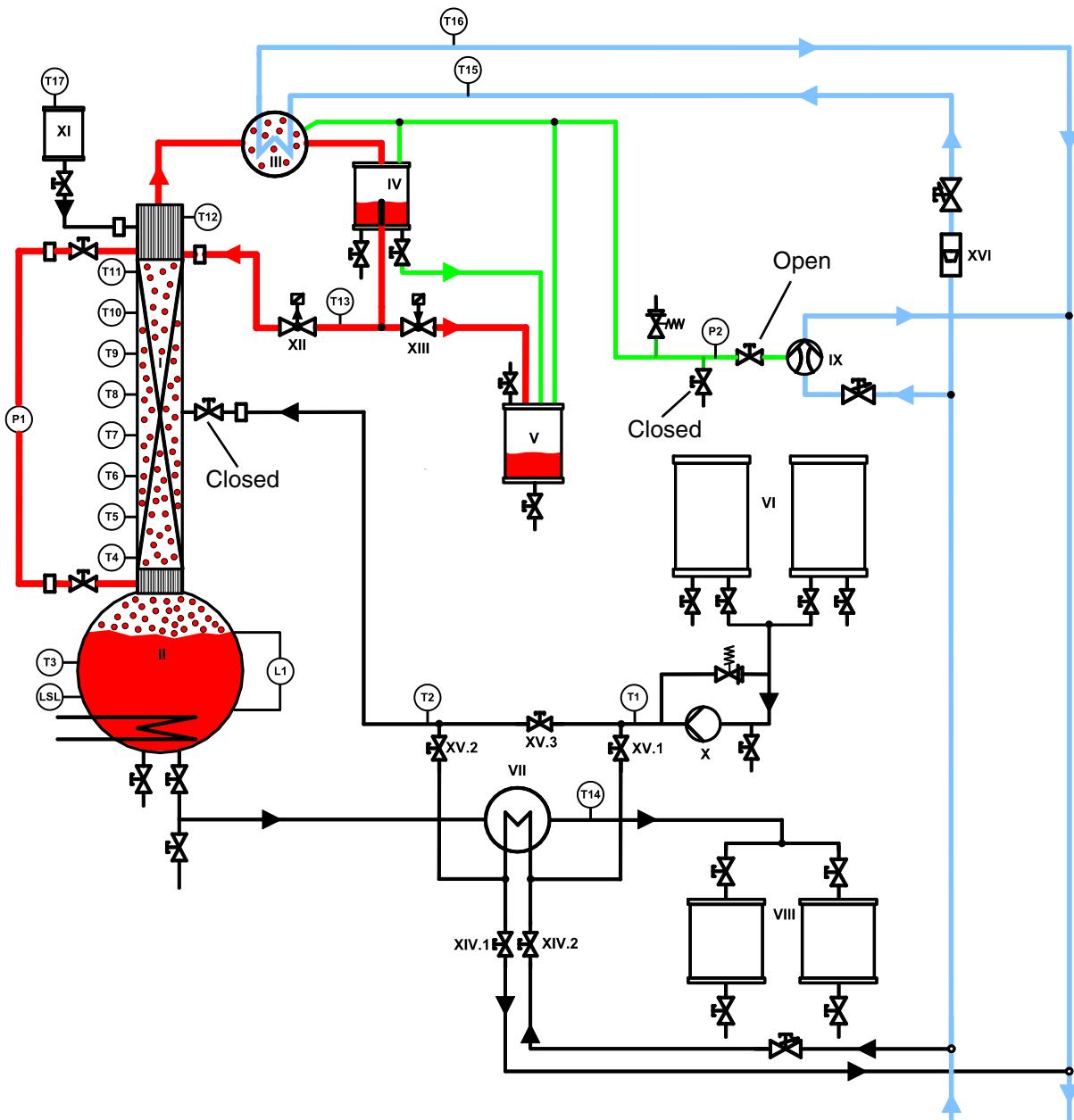


Fig. 5.13 Process diagram for a batch experiment under vacuum

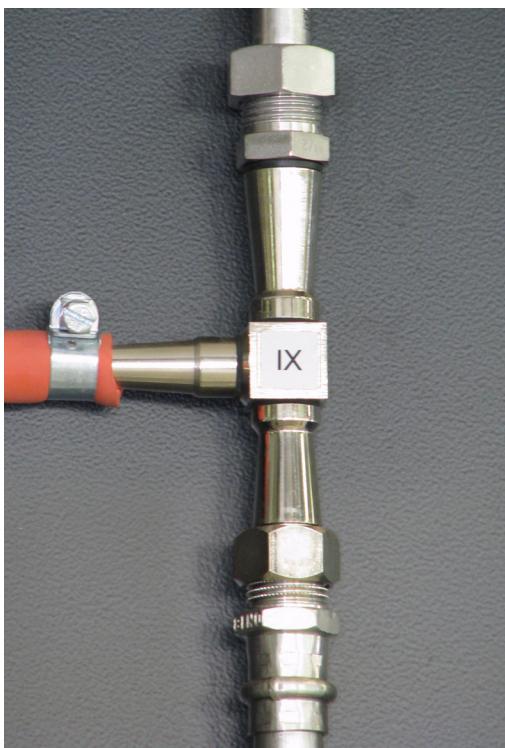
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Fig. 5.14 Glass filter pump

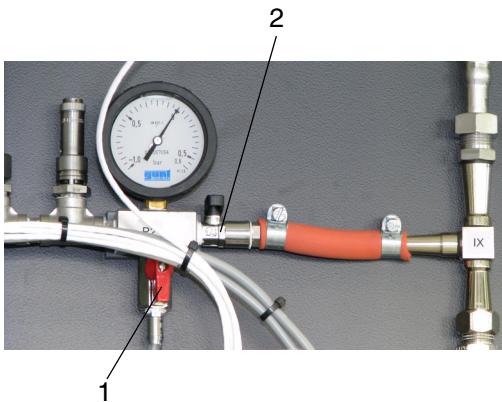


Fig. 5.15 Stop valves on the pressure gauge

The vacuum pump (IX) is executed as a glass filter pump - see Fig. 5.14 . When the water valve is opened the flow passes through the glass filter pump from bottom to top. The water jet effect causes air to be drawn in at the left side pump connection, producing a vacuum.

The vacuum can be influenced by altering the water flow. A higher vacuum is produced by increasing the water flow.

Take care to have reflux water flow by gravity (connection unpressurized; pressure in the reflux line could result in insufficient vacuum).

The process diagram Fig. 5.13 specifies the positions of the two small stop valves close to the pressure gauge. They are shown in the adjacent photo Fig. 5.15 (items 1 and 2).

For operation under vacuum it is important to close the manual stop valve (1) underneath the pressure gauge. This prevents the pressure being equalised with the atmospheric pressure. For the same reason, the stop valve must be closed for venting of the top product tank (V).

When the vacuum pump (IX) is in operation, the stop valve (2) shown in Fig. 5.15 between the pressure gauge and vacuum pump is opened so that air can be extracted from the system.

As already explained in chapter 5.4.1, page 39, the stop valve at the feed connection of the column should be closed so as to avoid unnecessary thermal loading on the hose.

5.5 Experiments - continuous mode

5.5.1 Notes on performing experiments

Here, too, a variant of the actual process diagram is used to illustrate the flow through the unit - see Fig. 5.16, page 45.

Before performing an experiment, the manual stop valves must be set to produce the flow shown during subsequent operation.

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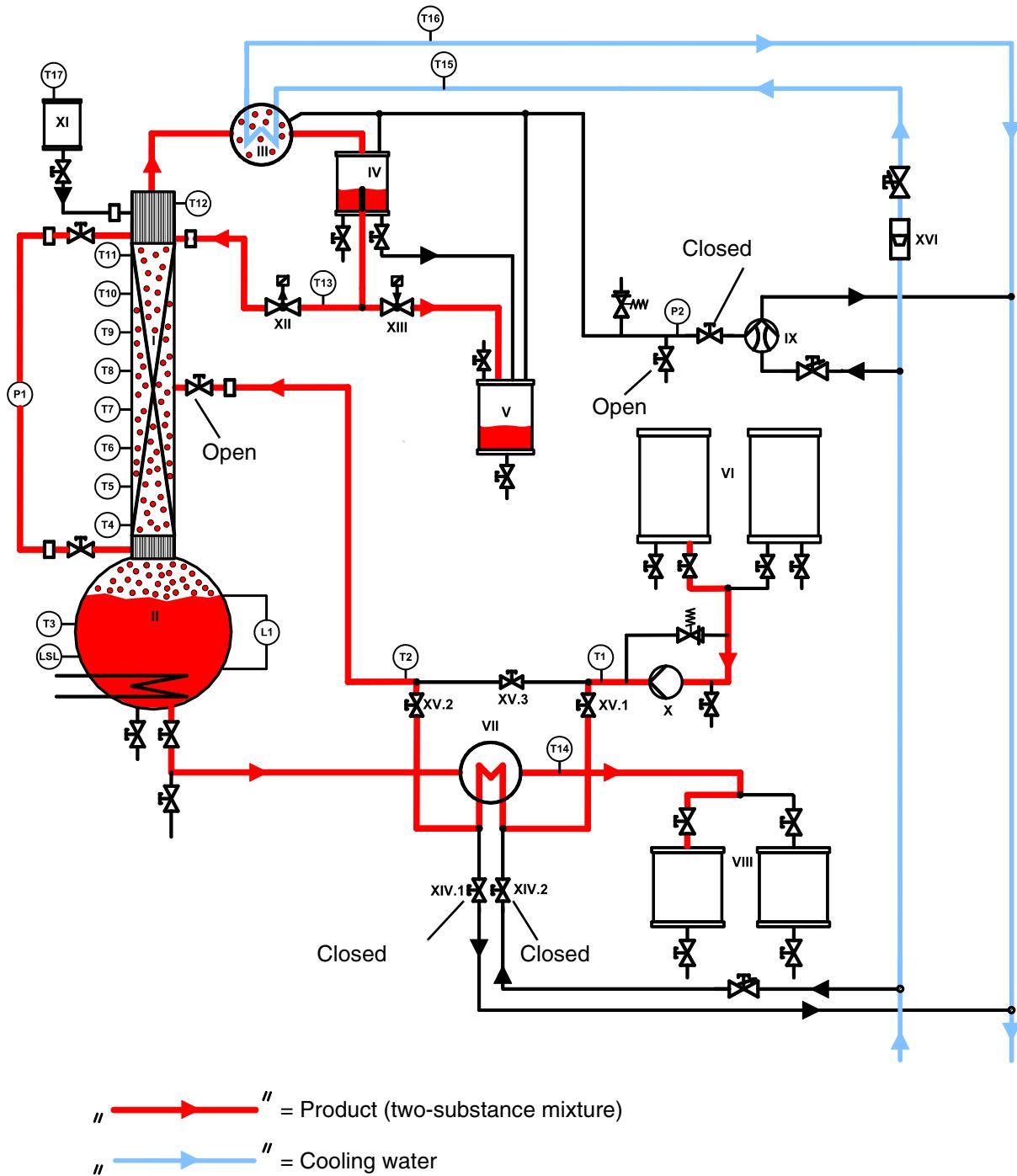


Fig. 5.16 Process diagram for continuous experiments at ambient pressure,
example with feed preheating

The positions of the stop valves close to the pressure gauge apply here as set out in chapter 5.4.1, page 39.

The stop valve at the column feed connection already mentioned initially remains closed. It is not opened until immediately before the feed pump is switched on.

Attention must also be paid to the following details:

1.) Tank breathing:

In continuous mode, the levels in the top product, feed and bottom product tanks change. To enable unhindered charging and draining of those tanks, they must be vented.

For the top product tank (V) this is done by opening the top stop valve.

For the feed and bottom product tanks it is necessary to unscrew the filler neck drain plugs. The drain plugs next to the filler necks are shown in the photos in Fig. 5.17.

2.) Bottom heat exchanger circuit:

The process diagram Fig. 5.16 also specifies the positions of the two stop valves (XIV.1) and (XIV.2). These two valves must be closed. If one of the valves is opened during operation, water will flow uncontrolled into the feed tank and the column.

It must also be ensured that the feed route from the tank into the column is open (see Fig. 5.16). To protect the pump, pumping against closed valves should be avoided.

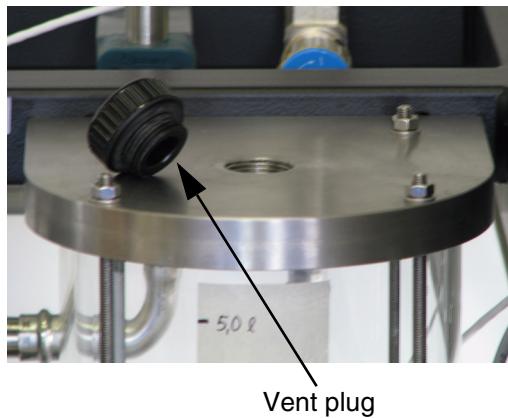


Fig. 5.17 Venting the feed tanks (VI)

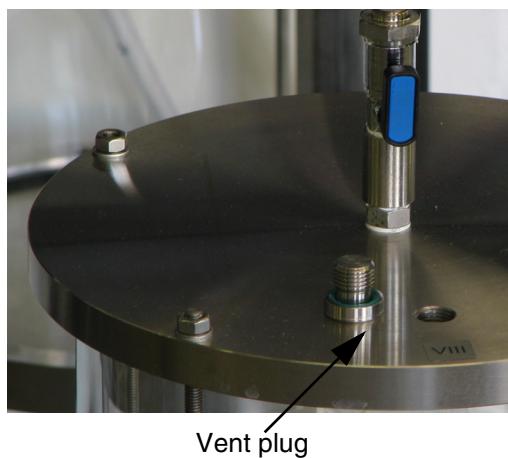


Fig. 5.18 Venting the bottom product tanks (VIII)

5.6 Practical tips

5.6.1 Requirements for the water / water hardness

To flush through the unit and for test purposes, water is generally used. Water is also a component of the recommended ethanol/water substance mixture.

The quality of the water used is one of the key factors determining the durability of the unit. The harder the water, the more deposits and scaling will form in the apparatus and the pipework. The behaviour of the unit will change even before the narrowest cross-section is blocked. This will impair the reproducibility of experiments.

Consequently, the use of water with a hardness $< 5^{\circ}\text{dH}$ is recommended for the cooling and motive water.

For the separation mixture we recommend using distilled water.

5.6.2 Pumping from the feed tanks into the evaporator

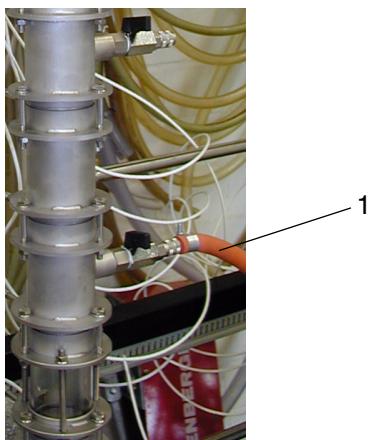


Fig. 5.19 Charging with sieve plate column

To charge the evaporator, the initial mixture is fed into the feed tanks. Where a sieve plate column is installed, it is advisable to connect the hose (1) to the bottom infeed connection of the column (see Fig. 5.19).

If it is connected to one of the higher fittings, the column may be flooded. The reason is that then, with the larger number of plates, the displaced air from below impedes the feed from above.

5.6.3 Bottom product cooling at the end of an experiment

At the end of an experiment there is bottom product at boiling temperature in the evaporator (II).

If there is enough time, the unit can be allowed to cool overnight with the bottom product in it. The next day the bottom product is then drained off so as to perform the outstanding measurements (mass, density, temperature).

If the bottom product is drained off immediately, however, there is a risk of scalding.



⚠ WARNING

Risk of scalding when removing the heated bottom product.

Use the facility to cool the bottom product (see below).

So what is needed is a method of cooling the entire evaporator content, also to save time and enable a second experiment to be conducted on the same day.

The bottom heat exchanger (VII) could, in principle, be used for cooling, if configured so that cooling water flows inside the jacket. However, the siphon in the evaporator prevents the remaining approximately 6l of bottom product from flowing through (see also chapter 6.2.1, page 60).

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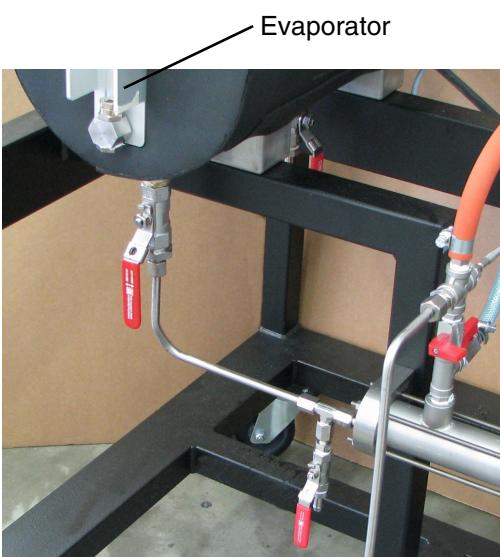
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Fig. 5.20 Evaporator without hose

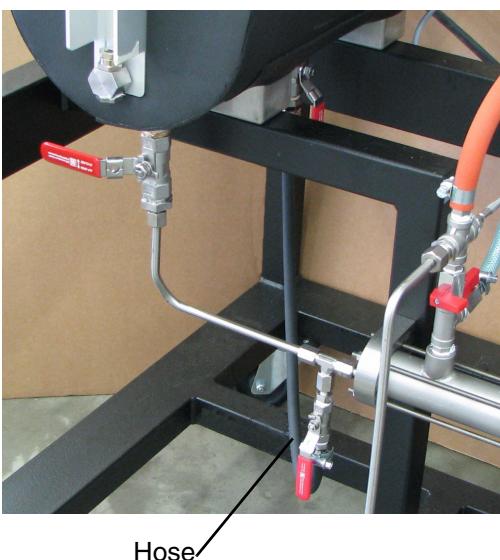


Fig. 5.21 Evaporator with hose,
for bottom product cooling
at end of experiment

An elegant solution to the problem is to “short-circuit” the siphon by a hose.

The evaporator without hose is shown in Fig. 5.20.

5.6.4 Reuse of input substances

It is possible to discard the top and bottom products after each experiment and apply a new mixture of input substances for each new experiment.

However, large quantities of input material can be saved by remixing product after performing an experiment. This mixture can be reused as the initial mixture for the subsequent experiment.

A certain loss of liquid while the experiment is being performed is inevitable (due to transfers, spillage when pouring, sampling, evaporation, and so on). Consequently, each new mixture should be composed of a larger initial mix quantity, so as to ensure there is adequate volume for each new experiment.

It is likely that the concentration of the mixture will change in the course of the experiments. The reason is that the more volatile substance evaporates and is lost to a greater extent. So the concentration should be checked every time prior to use. The mixture must be reset to the desired concentration as necessary.

5.6.5 Hose lengths

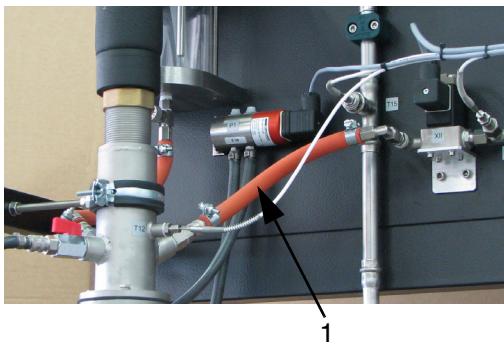


Fig. 5.22 Reflux hose (red)

Most of the lines interconnecting components of the CE 600 unit are pipes. It is, however, necessary to use hoses with couplings at some points.

This relates primarily to moving parts (such as the column head) or parts which can be removed and replaced (such as the column). It also relates to the alternative circuits (three infeeds into the sieve plate column).

Sagging of hoses, creating a sack effect, is not always avoidable. This does not usually entail any negative consequences. Problems do, however, arise if liquid collects in the “sack” and affects the process. This may happen at the following points:

- Reflux hose (item 1 in Fig. 5.22). Here liquid deposits may impair the flow of the top product completely (see also chapter 6.2.1, page 60, Reflux).
- Hoses between the column and the differential pressure sensor (items 2 and 3 in Fig. 5.23). Here liquid deposits may cause incorrect measurements.

The liquid may occur due to process events, such as flooding of the column. Often, though, it condenses from the system, during cooling after the unit has been in use.

It is advisable to detach the hoses in question and drain them before starting a new experiment. If problems persist, the hoses in question should be shortened to a minimum length.

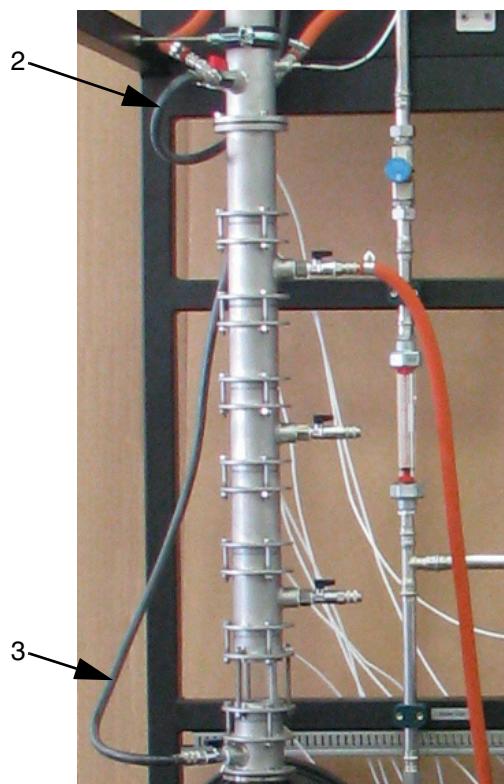


Fig. 5.23 Hoses for differential pressure measurement (black)

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6

Experiments - with ethanol/water mixture

All the experiments on which this section is based were conducted using an ethanol/water mixture.

The density was measured in order to determine the concentration. An areometer (measuring range 0,8 to 1,0kg/L), together with an upright cylinder (capacity 500ml) was used to measure the density. As a result, the sample volume has been specified and the number of possible samples limited.

This section occasionally refers to CE 600 components without showing the process diagram adjacent to the text. For clarification, please refer to the process diagram in Fig. 2.1, page 6.

6.1

Recommended ethanol/water mixture

An ethanol/water mixture is recommended for operation of the unit, for safety reasons.

6.1.1

Material values

The concentration of a material mixture can be determined from the mixture's density.

Knowledge of the densities of the input substances alone is not sufficient however, because the ratio between the concentration and the density of an ethanol/water mixture is not linear. The reason is that the ethanol and water molecules in the mixture influence each other and create additional binding forces (hydrogen bonding).

Consequently, the molecules may take up a smaller overall space, so the volume of the mixture is less than the sum of the individual input substance volumes (**volume contraction**).

In view of that, table values have to be applied across the entire concentration range.

The concentration of an ethanol/water mixture is not only dependent on the mixture density, but also the mixture temperature.

Tables are frequently to be found which set out the density and concentration for a fixed reference temperature. The reference temperature quoted is often 20°C, sometimes 15°C. Tables for reference temperatures above 20°C are difficult to find.

However, extensive material values are provided by the Organisation Internationale de Métrologie Légale (OIML) in the form of “International Alcoholometric Tables” (link: www.oiml.org). They set out the correlations between density and concentration for the reference temperatures -20 to +40°C, for both volumetric and mass fractions.

Extracted from those extensive material data listings, the following tables set out the densities, dependent on the ethanol mass fraction, for the temperatures +20 to +30°C.

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TABLE I

		$\varrho = \varrho(p, t)$									
p	t	21	22	23	24	25	26	27	28	29	30
0	998,20	997,99	997,77	997,54	997,29	997,04	996,78	996,51	996,23	995,94	995,65
1	996,11	995,88	995,64	994,40	993,15	992,89	992,62	992,34	992,05	991,75	991,50
2	994,49	994,27	994,05	993,52	993,32	993,06	992,78	992,50	992,21	991,90	991,62
3	992,73	992,50	992,05	991,50	991,55	991,28	991,00	990,72	990,42	990,12	990,33
4	991,02	990,81	990,58	990,34	990,09	989,83	989,56	989,28	988,95	988,69	988,38
5	989,18	989,15	988,92	988,68	988,42	988,15	987,88	987,59	987,30	986,99	986,68
6	987,18	987,15	987,06	986,80	986,53	986,25	985,95	985,65	985,34	985,02	985,02
7	986,24	986,20	985,17	985,49	985,22	984,94	984,65	984,36	984,05	983,73	983,40
8	984,73	984,49	984,23	983,96	983,68	983,40	983,10	982,79	982,48	981,82	981,82
9	983,27	983,01	982,15	982,47	982,18	981,89	981,58	981,26	980,94	980,60	980,26
10	981,85	981,58	981,30	980,71	980,41	980,09	979,76	979,43	979,08	978,73	978,73
11	980,46	980,18	979,89	979,58	979,27	978,95	978,63	978,29	977,94	977,59	977,22
12	979,10	978,80	978,50	978,18	977,86	977,53	977,18	976,83	976,47	976,11	975,73
13	977,16	977,13	976,80	976,50	976,45	976,12	974,72	974,35	973,97	973,58	973,02
14	976,44	976,11	975,78	975,43	975,08	974,72	973,33	972,94	972,55	972,15	972,78
15	975,13	974,19	974,44	974,08	973,71	973,33	972,95	971,54	971,13	970,74	971,32
16	973,83	973,47	973,11	972,73	972,34	971,95	970,56	970,15	969,71	969,29	969,85
17	972,44	972,16	971,77	971,38	970,97	970,56	969,60	969,17	968,73	968,28	968,38
18	971,74	970,44	970,02	969,60	969,22	968,17	968,73	968,29	967,83	966,37	966,90
19	969,93	969,51	969,19	968,66	968,22	967,31	966,85	966,38	965,90	965,41	965,41
20	968,61	968,17	967,73	967,28	966,82	966,35	965,88	965,40	964,91	964,41	963,91
21	967,27	966,81	966,35	965,88	965,40	964,91	964,42	963,92	963,42	962,90	962,38
22	965,90	965,43	964,94	964,45	963,96	963,45	962,94	962,43	961,90	961,37	960,83
23	964,51	964,02	963,51	963,01	962,99	962,49	961,97	961,44	960,90	960,36	959,81
24	963,53	962,05	961,53	961,03	960,99	960,45	959,91	959,35	958,79	958,23	957,66
25	961,63	960,56	960,56	960,02	959,46	958,91	958,34	957,77	957,19	956,61	956,02
26	960,14	959,19	959,03	958,47	957,90	957,32	956,74	956,16	955,56	954,96	954,36
27	958,61	958,04	957,47	956,89	956,30	955,71	955,11	954,51	953,90	953,28	952,66
28	957,05	956,46	955,88	954,66	954,05	953,44	952,82	952,20	951,57	950,93	950,28
29	955,44	954,83	954,22	953,61	952,99	952,36	951,73	951,10	950,46	948,82	948,17
30	953,78	953,16	952,54	951,91	951,27	950,63	949,99	949,34	948,69	948,03	947,37
31	952,19	951,46	950,82	950,17	949,52	948,87	948,21	947,55	946,88	946,21	945,53
32	950,36	949,05	948,39	947,71	947,73	947,06	946,39	945,72	945,04	944,35	944,04
33	948,58	947,92	947,25	946,58	945,90	945,22	944,54	943,85	943,16	942,47	941,77
34	946,77	946,19	945,41	944,73	944,04	943,35	942,65	941,95	941,25	940,54	939,83
35	944,92	944,23	943,54	942,84	942,14	941,44	940,73	940,02	939,31	938,59	937,87
36	943,92	942,33	941,63	940,92	940,21	939,49	938,78	938,07	937,33	936,61	935,88
37	941,11	940,40	939,68	938,96	938,24	937,52	936,79	936,06	935,33	934,59	933,86
38	939,16	938,43	937,71	936,98	936,25	935,51	934,78	934,04	933,30	932,55	931,81
39	937,16	936,43	935,70	934,96	934,22	933,48	932,74	931,99	931,24	930,49	929,73
40	935,15	934,41	933,66	932,92	932,17	931,42	930,67	929,92	928,40	927,64	927,37
41	933,10	932,35	931,60	930,85	930,10	929,34	928,58	927,82	927,06	926,52	926,31
42	931,03	930,38	929,22	928,76	928,00	927,24	926,47	925,70	924,93	924,21	923,38
43	928,04	928,18	927,41	926,65	925,88	925,11	924,34	923,56	922,79	922,01	921,43
44	926,42	926,06	925,29	924,52	923,74	922,96	922,41	921,19	919,62	919,84	919,05
45	924,69	923,92	923,14	922,36	921,58	920,80	920,02	919,23	918,44	918,65	918,05
46	922,33	921,76	920,98	919,41	918,62	917,83	917,04	916,25	915,45	914,65	914,06
47	920,37	919,58	918,80	918,01	917,22	916,43	915,64	914,84	913,04	912,24	911,47
48	918,18	917,39	916,60	915,81	915,02	914,22	913,42	912,62	911,82	910,01	909,21
49	915,98	915,19	914,40	913,60	912,80	912,00	911,20	910,39	909,59	908,78	907,96
50	913,77	912,98	911,38	910,57	909,77	908,96	908,15	907,34	906,53	905,71	905,50

Tab. 6.1 For the ethanol/water mixture: Density ϱ in kg/m³ as a function of the ethanol mass fraction p and temperature t , $0 \leq p \leq 50\%$, $20 \leq t \leq 30^\circ\text{C}$

CE 600

CONTINUOUS RECTIFICATION

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

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

Tab. 6.2 For the ethanol/water mixture: Density ρ in kg/m³ as a function of the ethanol mass fraction p and temperature t , $50 \leq p \leq 100\%$, $20 \leq t \leq 30^\circ\text{C}$

6.1.2 Determining concentration by density and temperature

The object of density measurement is to determine the concentration of the mixture.

Common concentrations quoted for the ethanol/water mixture are the ethanol volumetric fraction and mass fraction.

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

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

A comparison of the densities at different temperatures shows that the concentration is substantially dependent on the sample temperature as well as the density.

If possible, the samples should be appropriately **temperature-controlled** prior to measuring the density, such as to the reference temperature 20°C. Then the concentration can be read directly from the table with the measured density at the relevant temperature.

No such temperature control was available for the experiments on which this chapter is based. Consequently, the density and temperature of the sample were measured for each concentration calculation. Allowance is made for the influence of the temperature by means of linear interpolation (see Evaluation, chapter 6.3.5.2, page 80 ff).

The major influence of small changes in density on the ethanol mass fraction is illustrated in chapter 6.3.6, page 87 ff. under item 5., based on a concrete example.

6.1.3 Mixing behaviour

Ethanol and water are mutually soluble. Solubility exists across the complete concentration range.

Nevertheless, **mixing problems** are to be expected!

Example: At the end of the experiment approximately 8L of mixture remain in the CE 600 evaporator, with 20%*m* ethanol, temperature 90°C. When the experiment is complete the top products are collected and fed by way of a feed tank into the evaporator, 2L, 90%*m* ethanol, 25°C.

This mixture remains in the evaporator overnight.

Result the next morning: The stratification in the insulated evaporator is retained. There are still major differences in temperature, density and concentration between the mixture at the top and bottom of the evaporator!

Result: To determine the density and temperature of the mixture, the entire evaporator content must be drained off and mixed.

6.2 Definition of background conditions, experiment series

As already described in chapter 5.3, page 38, there is a wide variety of experiment variants, with different background conditions. This also applies to the ethanol/water mixture.

Depending on the object of the experiment, a distinction can be made between three different categories of usage:

1. Spontaneous experiments, with frequently changing conditions
2. Automatic mode, using the various control loops
3. Comparative experiments, recording the influence of a change

Re 1. Spontaneous experiments: Spontaneous experiments rapidly convey a feel for the unit and for the material mixture. While the unit is running, immediately after altering a condition, the reaction of the unit is observed (change in temperatures, differential pressure, boiling behaviour, ...). The limits of the unit are plotted. An estimation can be quickly obtained as to whether stable operation is to be expected under specific conditions.

Re 2. Automatic mode: Operating the unit in automatic mode is useful when the object of the experiment is to prepare for practical production operations. The behaviour of the various control loops can be observed. And the associated measured values can be recorded. The reaction to changes in control parameters can be investigated. Two different temperature control systems are also possible here.

Either the temperature T3 in the evaporator or the temperature T12 in the column head can be controlled.

Re 3. Comparative experiments: The object of comparative experiments is to determine the influence of one change on the measurement results. To do this, the individual experiments must be reproducible. Reproducibility is improved when disturbances to the experiment are minimised. Consequently, there are advantages in avoiding automatic mode when performing comparative experiments. Instead fixed values should be used for the heater and for the reflux ratio.

These instructions primarily relate to comparative experiments.

6.2.1 Method of operation of the unit

In contrast to large-scale production units, the CE 600 is not optimised for a specific application. Rather, attention was paid to covering a large number of different experiment variants at reasonable expense, including in terms of the control engineering.

The restricted height means that some infeed heads and supply pressures are limited.

Another factor is the interaction of thermal and hydraulic influences throughout the system.

In spontaneous experiments, changes sometimes do not lead to the intended result.

So at this point the **method of operation of the unit for an ethanol/water mixture** is depicted:

- **Column differential pressure:** The differential pressure is a measure of the boiling intensity and separation efficiency in the column. We recommend setting the heater to produce a differential pressure of around 10 to 20mbar. If the pressure falls below 10mbar, the boiling intensity is low and the experiment takes an unnecessarily long time. If the pressure exceeds 20mbar the boiling intensity is high. The volume of steam in the column is then sometimes so great that the downward flow of condensate is too severely impaired. The result is then unstable operation.
- **Liquid level in evaporator (II) in batch experiments:** The minimum is given by the switching point of the level switch (at just over 3L). For safety reasons, the heater is switched off if the level falls below the minimum.
The maximum is based on preventing flooding of the evaporator. The interface between the liquid and the steam/air should always remain visible in the level indicator tube. This results in the practical maximum of around 10L.
- **Liquid level in evaporator in continuous experiments:** A siphon is installed in the evaporator upstream of the outlet.

If the siphon was not installed, the evaporator would quickly be completely drained as the flow passes from the evaporator to the bottom product tank (VIII).

The driving force in this is not only the liquid head at the outlet, but also the higher operating pressure in the evaporator, referred to the ambient pressure in the bottom product tank. If the level falls below about 6L, the siphon restricts the flow from the evaporator to the bottom product tank at ambient pressure. This means the range for the usable volume at ambient pressure is limited to 6 to 10L. This requires careful observation of the evaporator level during operation and regular adjustment by adjusting the position of the stop valve at the evaporator outlet.

However, the range for the usable volume is extended during operation in accordance with the prevailing operating pressure.

- **Reflux:** The reflux flows when the valve (XII) is open owing to the difference in height between the phase separation tank (IV) and the column head. This difference in height must overcome the pressure difference built up between the column head and the phase separation tank during operation (ambient pressure). If the pressure difference is too large the reflux will be impaired, or even prevented altogether. Conversely, the flow from the phase separation tank to the top product tank (V) is not impeded when the valve (XIII) is open (both tanks are at ambient pressure). In the case of high reflux ratios it is even possible that top product may back-up in the phase separation tank.
In such cases this pressure difference should be reduced, such as by reducing the heat output.

- **Vacuum mode:** During operation, minor changes in pressure have a significant influence on the boiling temperature. By changing the pressure a few hundredths of a bar, the boiling intensity in the column can be greatly increased, or may virtually disappear entirely. This in turn influences the pressure in the system. Regulating the pressure by altering the water flow into the vacuum pump (IX) requires some practice. So CE 600 enables relatively stable operation in vacuum mode during batch experiments. However, vacuum mode in continuous experiments cannot run permanently stable because the overall system responds too sensitively to fluctuations.
- **Infeed heads of the sieve plate column:** The choice of infeed to the sieve plate column influences the stability and reproducibility of the experiment. Top infeed can result in inadequate stability and uniformity.
- Production factors, fluctuations in ambient conditions and changes during operation of the unit may cause the unit properties to change and so shift the optimum **parameter over time.**

6.2.2 Comparative experiments, experiment series

When defining the conditions for comparative experiments and experiment series, the difficulty is in safeguarding continuous, stable and reproducible operation for all the planned experiments. Without practical testing, there is a severe risk that any unsuitable choice of conditions will only be identified during the actual running of the experiments. That is why it is advisable to check a planned new series of experiments in advance by means of spontaneous experiments and correct them as necessary.

It is conceivable that a large number of different experiment series could be developed over time. In the following chapters two experiment series are presented in which the conditions of the individual experiments are coordinated.

It is possible to discard the top and bottom products after each experiment and apply a new mixture of input substances for each new experiment.

However, for the experiments on which this chapter is based the products were remixed after each experiment (see also chapter 5.6.4, page 50). The experiment reports, worksheets, analyses and suchlike detailed in the following relate to this method.

6.2.3 Example of a batch experiment series

The batch experiment series comprises experiments V1 to V5.

Tab. 6.3 lists the common conditions applicable to all experiments V1 to V5.

The heating power is indicated as a percentage of the maximum 4,0kW, in line with the input prompt on the PC.

Condition type	Name	Symbol/unit	Value
Initial mixture	Volume	V in L	10
	Ethanol mass fraction	w_E in %m	25
Cooling water quantity	Volumetric flow	\dot{V} in L/h	150
Start with	Reflux ratio	R in %	100
	Heating power	P_{el} in %	100
At $T_3 \geq T_{Warm}$, Continue with	Reflux ratio	R in %	100
	Heating power	P_{el} in %	20
At $T_x = \text{constant}$, operation with	Reflux ratio	R in %	50
	Heating power	P_{el} in %	30
At $T_3 \geq T_{End}$, Cancel with	Reflux ratio	R in %	50
	Heating power	P_{el} in %	0
After $t = 5 \text{ min}$, sampling	Reflux ratio	R in %	100
	Heating power	P_{el} in %	0

Tab. 6.3 Common conditions for batch experiment series

The “variable” conditions by which experiments V1 to V5 differ are set out in Tab. 6.4.

Condition type	Symbol, Unit	Experiment				
		V1	V2	V3	V4	V5
Column type: Pd, SP-8, SP-4		SP-8	SP-8	SP-8	Pd	SP-4
Operation at ambient pressure or vacuum	p = atm or p in bar abs.	atm	0,5	atm	atm	atm
Reflux ratio, in operation	R in %	0	0	50	50	50
Evaporator temperature, after warm-up	T_{Warm} in °C	70	50	70	70	70
Evaporator temperature, at end of operation	T_{End} in °C	92	75	92	92	92

Key:

Pd: Packed column

SP-8: Sieve plate column with 8 plates

SP-4: Sieve plate column with 4 plates

atm: Atmospheric pressure / Ambient pressure

Tab. 6.4 Variable conditions for batch experiment series

After performing these five experiments, the following comparisons are possible:

- Sieve plate column / Packed column
- Sieve plate column, 8 plates / Sieve plate column, 4 plates
- Vacuum mode / Operation at ambient pressure
- Reflux ratios 0, 50 and 100%

Experiment V3 is described in detail and evaluated by way of example for this experiment series in chapter 6.3, page 70.

The abort condition with " T_{End} = Evaporator temperature, at end of operation, designated in the table as $T_3 \geq T_{End}$ requires some explanation.

It is necessary to adjust the abort condition because the boiling points of the input substances and mixtures are pressure-dependent. As the absolute pressure falls the boiling points drop. An identical abort temperature for experiments in vacuum mode and at ambient pressure would make it difficult to compare the experiments.

In fact, an unchanged abort temperature of 92°C would make no sense for the experiment V2 described. Then the abort temperature would be greater than the boiling temperature of the high-boiling water component (around 81°C at 0,5 bar abs.). That means 92°C in the evaporator cannot be reached at 0,5 bar abs.

That is why the numeric values for the abort temperature T_{End} are defined here as the mean value of the initial mixture boiling temperature and the boiling temperature of pure water, referred to the respective pressure.

Similarly with regard to the feed-forward condition "Evaporator temperature, after warmup", designated in the tables as T_{Warm} . These temperatures, too, are selected so that the experiments are roughly comparable for vacuum mode and operation at ambient pressure (temperature gap between reduced heating power after warm-up and attainment of boiling temperature).

6.2.4 Example of a continuous experiment series

The continuous experiment series comprises experiments V6 to V8.

Tab. 6.5 lists the common conditions applicable to all experiments V6 to V8.

Condition type	Name	Symbol/unit	Value
Initial mixture in the evaporator	Volume	V in L	9
	Ethanol mass fraction	w_E in %m	25
Initial mixture in the feed tanks	Volume	V in L	10
	Ethanol mass fraction	w_E in %m	25
Cooling water quantity	Volumetric flow	\dot{V} in L/h	300
Start with	Reflux ratio	R in %	100
	Heating power	P_{el} in %	100
At $T_3 \geq 80^\circ\text{C}$, Continue with	Reflux ratio	R in %	100
	Heating power	P_{el} in %	25
5 min after first reflux, operation with	Reflux ratio	R in %	25
	Heating power	P_{el} in %	25
	Start feed pump	f in %	20
	Start B.pr. in B.pr.tank	-----	----
After feed volume 5L, abort with	Reflux ratio	R in %	25
	Heating power	P_{el} in %	0
	Stop feed pump	f in %	0
	Stop B.pr. in B.pr.tank	-----	----
After $t = 5$ min, sampling	Reflux ratio	R in %	100

Key:

B.pr.: Bottom product

B.pr.tank: Bottom product tank

T3: Evaporator temperature

Tab. 6.5 Common conditions for continuous experiment series

The heating power is indicated as a percentage of the maximum 4,0kW, in line with the input prompt on the PC.

For the feed pump, the frequency of the applied current is indicated as a percentage of the maximum frequency.

Experiments V6 to V8 are intended for operation at ambient pressure. The sieve plate column is used in each, to enable comparisons with the batch experiments V1 to V3.

The abort condition “After feed volume 5L” is a compromise between the time and measurement commitment and the additional expected results. If required, the experiment can of course be prolonged by likewise pumping the remaining 5L feed into the column.

The “variable” conditions by which experiments V6 to V8 differ are set out in Tab. 6.6.

Condition type	Experiment		
	V6	V7	V8
Feed preheating Yes/No	No	Yes	Yes
Infeed to sieve plate column Centre/Bottom	Bottom	Bottom	Centre

Tab. 6.6 Variable conditions for continuous experiment series

Experiment V7 is described in detail and evaluated by way of example for this experiment series in chapter 6.4, page 93 ff.

6.3 Example of a batch experiment, V3

Experiment V3 originates from the batch experiment series (see also chapter 6.2.3, page 65). **Conditions** for this experiment are:

- Initial mixture 10L, ethanol fraction 25% m
- Sieve plate column
- Operation at ambient pressure
- Cooling water flow 150L/h
- Operation at heating power 30%,
Reflux ratio 50%
- Rectification aborted (heater off) on reaching
evaporator temperature T3 = 92°C

6.3.1 Experiment aims

Experiment aims are:

1. To concentrate the ethanol
2. To record the measured value using the data acquisition program (temperature and pressure profiles)
3. To determine the ethanol concentration in the initial mixture, top product and bottom product
4. To depict the change over time of the ethanol concentration in the top product
5. To check the concentrations by means of mass balances

6.3.2 Experimental setup

Connected CE 600 unit, with accessories as set out in chapter 5.2, page 37.

6.3.3 Performing the experiment

- Observe the safety instructions (see chapter 3, page 19)
- Prepare the unit as set out in chapter 5.4.1, page 39. Open the manual stop valve underneath the pressure gauge.
- The requirement is a canister containing more than 10L of initial mixture, ethanol: 25% m , water: 75% m (new addition, e.g. for 12L, with 2885g ethanol and 8655g water).
- Mix the initial mixture in the canister.
- Identify the density and temperature of the initial mixture. Check the concentration.
- Transfer the initial mixture from the canister into the two feed tanks (VI). Use measuring cups for this, and weigh the content of each cup (net weight of each). Fill the feed tanks up to their 5 litre marks. Also weigh the residue in the measuring cup.
- Transfer the initial mixture from the feed tanks through the column into the evaporator (see chapter 5.6.2, page 47). To do so, open the stop valve on the column during pumping.
- Press Reset on the differential pressure display (item 23 in Fig. 2.6, page 10).
- Unit operation by PC. Make the settings for data acquisition, open the measured value file (see chapter 2.4.2, page 13).
- Set the reflux ratio to 100% initially
- Open the condenser water supply and adjust it to about 150L/h
- Switch on the heater. Initial fixed value 100%

- When evaporator temperature $T_3 = 70^\circ\text{C}$ is reached, reduce the heater to the fixed value 20%
- Wait for distillate to appear at the phase separation tank (IV), reflux begins and the system reaches a state of equilibrium (temperatures constant about 20 minutes after first appearance of distillate).
Then changes to operating conditions:
 - Heater from 20 to 30%
 - Reflux ratio from 100 to 50%
- When the minimum sample volume is reached, withdraw the top product from the top product tank (V). Determine the mass, density and temperature. Keep the sample (for a later mixed sample). Perform a new measurement as soon as the minimum sample volume has been reached again.
- Abort on reaching $T_3 = 92^\circ\text{C}$
- 5 minutes after aborting, change reflux ratio to 100%. Shut off the water supply.
- Measure the remaining top product (residue from top product tank together with residue from phase separation tank); determine the mass, density and temperature.
- Pour together and intermingle the previous top product samples and measure the total mass, density and temperature.
- Cool the bottom product from the evaporator (see also chapter 5.6.3, page 48). Drain off the cooled bottom product, weigh it and mix it in the second canister.
Take a sample of the bottom product mixture, determine density and temperature.

- Fill canister with unused initial mixture and with top and bottom product. Mix well.
This provides a new initial mixture for the next experiment.

6.3.4 Measured values

6.3.4.1 Measurement data from the data acquisition program (experiment aim 2)

Using the data acquisition program, the measured values were saved in “.dat” file format. This **measured value file** contains a chronological sequence of measured data records. A **measured data record** is a snapshot of all the measured values at the given point in time (see also chapter 2.4.2, page 13).

For this experiment an interval of 30 seconds was selected (representing the time between recording of two measured data records). Based on a minimum recording period of 100 minutes, this produces more than 4200 measured values (two measured data records per minute, each with 21 measured values).

A tabular representation of the complete measured value file, with all measured data records, would be too extensive to set out at this point. Consequently, a selection was made to depict the most informative time bands.

The measured values for the selected time bands are imported into MS-Excel (see measured value tables Tab. 6.7 to Tab. 6.9). The MS-Excel Import function is used to import the measured values.

The selected time bands are:

- Column start-up until production of the first distillate
- Start of rectification under operating conditions
- End of rectification under operating conditions

Alongside the differential pressure P1 and the temperatures T1 to T16, these measured value tables also contain the three control variables Y1 to Y3. In the experiment described here, fixed values were programmed on PC rather than using the software controllers. Using the defined operating conditions (heater $Y1=25\%$ and reflux ratio $Y2=50\%$), this enables the start and end of the actual experiment (here 14:30 and 15:06 hours) to be determined from the measured value tables.

The differential pressure and selected temperatures are presented graphically in chapter 6.3.5.1, page 78.

CE 600**CONTINUOUS RECTIFICATION**

t hh:mm:ss	T1 °C	T2 °C	T3 °C	T4 °C	T5 °C	T6 °C	T7 °C	T8 °C	T9 °C	T10 °C	T11 °C	T12 °C	T13 °C	T14 °C	T15 °C	T16 °C	P1 mbar	Y1, P _{el} %	Y2, R %	Y3, f %		
13:31:17	23.5	23.6	26.1	24.0	23.7	24.5	25.3	25.9	26.0	25.9	26.1	26.1	24.2	22.5	23.4	24.4	-0.1	0	0	0		
13:55:47	22.6	23.4	82.5	59.1	30.5	24.6	24.4	25.2	25.1	25.2	25.2	24.0	22.8	14.9	15.2	-0.1	20	100	0	0		
13:56:17	22.6	23.4	82.8	60.1	31.0	24.2	24.4	25.0	25.0	25.2	25.2	24.0	22.8	14.6	15.2	-0.1	20	100	0	0		
13:56:47	22.5	23.4	83.2	59.8	31.5	24.6	24.2	25.0	25.1	25.2	25.2	24.0	22.6	14.7	15.3	-0.1	20	100	0	0		
13:57:17	22.6	23.4	83.3	61.8	32.4	24.7	24.3	25.0	25.1	25.2	25.1	24.0	22.7	14.8	15.2	-0.1	20	100	0	0		
13:57:47	22.6	23.4	83.8	61.3	33.4	24.8	24.5	25.3	25.0	25.2	25.1	24.0	22.8	14.7	15.0	0.0	20	100	0	0		
13:58:17	22.6	23.4	84.0	62.1	33.8	24.7	24.5	24.8	25.1	25.1	25.1	24.0	22.7	14.8	15.2	0.0	20	100	0	0		
13:58:47	22.6	23.4	84.4	62.9	34.6	24.7	24.4	25.0	25.0	25.2	25.0	24.0	22.9	14.7	15.1	-0.2	20	100	0	0		
13:59:17	22.6	23.4	84.7	69.8	35.9	24.8	24.5	25.0	25.0	25.1	25.3	24.0	22.9	14.7	15.2	0.0	20	100	0	0		
13:59:47	22.6	23.4	84.9	72.4	36.4	24.9	24.3	24.9	25.0	25.2	25.1	24.0	22.8	14.6	15.1	0.1	20	100	0	0		
14:00:17	22.6	23.4	85.4	71.9	34.2	25.0	24.4	25.0	25.0	25.0	25.0	24.0	22.8	14.8	15.2	0.2	20	100	0	0		
14:00:47	22.5	23.4	85.3	74.0	32.7	24.9	24.4	25.0	25.0	25.1	25.0	25.0	24.0	22.8	14.7	15.2	0.1	20	100	0	0	
14:01:17	22.6	23.4	85.9	75.1	33.6	25.0	24.4	25.0	25.0	25.1	25.0	25.0	24.0	22.9	14.7	15.2	0.0	20	100	0	0	
14:01:47	22.6	23.4	85.9	76.2	43.7	25.3	24.3	24.8	25.0	25.1	25.0	25.0	24.1	22.9	14.7	15.2	0.2	20	100	0	0	
14:02:17	22.6	23.4	86.2	76.8	58.3	25.4	24.5	25.0	25.0	25.0	25.0	25.0	24.0	22.8	14.9	15.2	-0.1	20	100	0	0	
14:02:47	22.5	23.5	86.6	74.8	70.0	26.4	24.5	24.9	24.9	25.0	25.0	25.1	24.0	22.9	14.8	15.2	0.0	20	100	0	0	
14:03:17	22.6	23.4	86.8	80.3	68.3	33.6	24.7	24.9	24.9	24.9	24.9	25.0	24.0	22.9	14.8	15.2	0.0	20	100	0	0	
14:03:47	22.6	23.5	86.9	81.2	77.3	56.5	28.0	25.4	25.0	25.0	25.0	24.9	24.0	22.8	14.7	15.2	0.1	20	100	0	0	
14:04:17	22.6	23.4	86.9	80.4	79.4	67.7	33.3	26.2	25.0	25.0	25.0	24.8	24.0	22.9	14.9	15.3	1.0	20	100	0	0	
14:04:47	22.6	23.5	86.8	80.3	79.6	72.4	57.0	34.7	24.9	25.0	25.0	25.0	24.0	22.8	14.9	15.5	1.3	20	100	0	0	
14:05:17	22.6	23.5	87.2	80.7	80.0	78.7	66.2	37.4	24.9	24.9	25.0	25.0	23.9	22.9	14.8	15.3	1.7	20	100	0	0	
14:05:47	22.6	23.5	87.2	80.9	80.1	78.7	71.6	62.0	25.0	25.0	24.8	24.9	23.9	22.8	14.7	15.4	2.6	20	100	0	0	
14:06:17	22.6	23.5	87.2	81.0	80.3	79.2	77.6	73.2	25.1	25.0	25.0	24.9	23.9	22.8	14.9	15.3	2.9	20	100	0	0	
14:06:47	22.5	23.4	87.1	81.2	80.3	79.3	77.9	64.5	25.0	25.0	25.0	25.0	23.9	22.9	14.9	15.4	3.8	20	100	0	0	
14:07:17	22.6	23.4	87.2	81.4	80.6	79.5	78.3	78.0	75.9	25.0	25.0	25.0	24.9	23.9	22.8	14.9	15.3	4.2	20	100	0	0
14:07:47	22.6	23.5	87.2	81.5	80.7	79.6	78.5	78.2	74.9	52.1	52.1	50.0	24.0	23.1	14.9	15.4	5.4	20	100	0	0	
14:08:17	22.3	23.5	87.3	81.7	80.8	79.8	78.7	78.7	78.1	63.9	25.2	25.0	24.9	22.9	14.9	15.2	5.5	20	100	0	0	
14:08:47	22.6	23.5	87.2	81.7	80.9	79.9	78.8	78.7	78.4	75.3	56.2	25.2	24.9	22.9	14.7	15.2	6.1	20	100	0	0	
14:09:17	22.6	23.6	87.3	81.8	80.9	80.1	78.9	78.9	78.7	77.6	70.2	25.3	23.9	22.9	14.9	15.2	7.1	20	100	0	0	
14:09:47	22.5	23.5	87.4	82.1	81.2	80.0	79.1	79.1	78.7	78.1	65.5	65.2	23.9	22.8	14.9	15.4	7.8	20	100	0	0	
14:10:17	22.5	23.4	87.4	82.0	81.1	80.2	79.4	79.2	79.0	78.4	76.4	73.5	24.0	22.9	14.9	15.3	8.1	20	100	0	0	
14:10:47	22.6	23.4	87.4	82.3	81.4	80.3	79.2	79.4	79.2	78.5	77.6	77.3	23.8	22.9	14.9	15.3	8.4	20	100	0	0	
14:11:17	22.6	23.4	87.5	82.3	81.4	80.1	79.4	79.4	79.2	78.4	77.6	77.5	24.0	22.9	14.8	15.4	8.9	20	100	0	0	
14:11:47	22.6	23.5	87.5	82.9	81.5	80.2	79.3	79.5	79.3	78.8	77.7	77.7	23.9	22.9	14.9	15.3	9.6	20	100	0	0	
14:12:17	22.6	23.5	87.5	82.3	81.5	80.3	79.3	79.5	79.2	79.0	78.2	78.0	23.9	22.9	14.8	15.3	9.9	20	100	0	0	
14:12:47	22.6	23.6	87.6	82.8	81.7	80.4	79.3	79.5	79.5	78.7	78.6	78.6	23.9	22.9	14.7	16.1	9.6	20	100	0	0	
14:13:17	22.6	23.5	87.5	83.0	82.0	80.4	79.5	79.5	79.5	78.9	78.8	78.8	23.9	22.9	14.8	16.7	10.3	20	100	0	0	
14:13:47	22.6	23.5	87.6	83.6	82.2	80.4	79.4	79.4	79.5	79.4	79.1	79.0	24.0	22.9	14.8	17.1	10.0	20	100	0	0	
14:14:17	22.6	23.7	87.8	83.6	82.4	80.6	79.6	79.6	79.6	79.5	79.3	79.2	23.9	22.9	14.9	17.3	10.0	20	100	0	0	
14:14:47	22.7	23.4	87.7	83.6	82.6	80.6	79.6	79.6	79.7	79.5	79.5	79.0	24.0	22.9	14.9	17.2	9.9	20	100	0	0	

Temperatures and differential pressure according to flow diagram / system diagram.
 Y1, Y2, Y3: Controller Outputs for heater, Reflux ratio, frequency of pump drive.

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t hh:mm:ss	T1 °C	T2 °C	T3 °C	T4 °C	T5 °C	T6 °C	T7 °C	T8 °C	T9 °C	T10 °C	T11 °C	T12 °C	T13 °C	T14 °C	T15 °C	P1 mbar	Y1, P _{el} %	Y2, R %	Y3, f %	
14:28:47	22,6	23,7	88,4	86,8	85,2	82,3	80,6	80,5	80,2	79,8	79,7	79,4	25,5	23,1	14,8	17,5	20	100	0	
14:29:17	22,6	23,6	88,5	86,8	85,5	82,3	80,6	80,6	80,2	80,0	79,7	79,5	25,6	23,0	14,7	17,6	20	100	0	
14:29:47	22,7	23,7	88,5	86,9	85,5	82,2	80,5	80,3	80,3	80,0	79,7	79,4	25,6	23,0	14,7	17,5	16,2	20	100	0
14:30:17	22,7	23,6	88,5	86,9	85,1	81,9	80,5	80,3	80,2	79,9	79,8	79,5	25,6	23,1	14,8	17,4	16,5	20	50	0
14:30:47	22,6	23,7	88,5	86,9	85,8	83,1	81,2	80,8	80,3	80,1	79,9	79,7	25,3	23,1	14,7	19,3	15,0	30	50	0
14:31:17	22,6	23,7	88,5	87,7	86,1	84,3	81,7	81,0	80,5	80,2	80,0	79,7	25,3	23,1	14,7	19,6	14,2	30	50	0
14:31:47	22,6	23,6	88,7	87,7	86,9	85,0	82,7	81,4	80,5	80,2	80,0	79,8	25,2	23,1	14,7	19,7	14,5	30	50	0
14:32:17	22,6	23,7	88,8	87,5	87,2	85,5	83,3	81,7	80,6	80,3	80,1	79,8	25,0	23,0	14,7	19,8	13,5	30	50	0
14:32:47	22,6	23,7	88,7	87,9	87,4	86,1	83,9	82,2	80,6	80,3	80,1	79,8	25,0	23,1	14,7	20,0	14,0	30	50	0
14:33:17	22,5	23,6	88,8	88,0	87,4	86,6	84,2	82,5	80,9	80,4	80,1	79,8	25,1	23,1	14,7	20,0	14,0	30	50	0
14:33:47	22,6	23,8	88,8	88,0	87,6	86,6	84,6	82,8	81,0	80,4	80,2	80,0	25,1	23,1	14,6	20,1	14,3	30	50	0
14:34:17	22,6	23,8	88,9	87,9	87,7	86,6	84,8	83,0	81,1	80,4	80,2	80,0	25,3	23,0	14,6	20,1	13,9	30	50	0
14:34:47	22,6	23,7	88,9	88,0	87,5	86,9	85,1	83,2	81,1	80,4	80,3	80,2	25,3	23,0	14,8	20,1	14,5	30	50	0
14:35:17	22,6	23,8	89,0	88,1	87,8	87,0	85,1	83,3	81,2	80,6	80,3	80,1	25,6	23,1	14,6	20,3	13,8	30	50	0
14:35:47	22,6	23,7	89,0	88,1	87,8	86,9	85,1	83,5	81,2	80,7	80,1	80,1	25,9	23,1	14,6	20,2	13,8	30	50	0
14:36:17	22,6	23,7	89,0	88,1	88,0	86,9	85,3	83,6	81,2	80,6	80,3	80,2	26,3	23,1	14,7	20,2	14,4	30	50	0
14:36:47	22,6	23,6	89,1	88,0	87,8	87,0	85,1	83,6	81,3	80,6	80,3	80,2	26,4	23,1	14,7	20,4	14,3	30	50	0
14:37:17	22,6	23,8	89,1	88,3	87,7	87,2	85,2	83,6	81,2	80,6	80,3	80,6	26,5	23,0	14,7	20,4	14,6	30	50	0
14:37:47	22,6	23,7	89,3	88,3	87,9	87,1	85,4	83,4	81,4	80,9	80,6	80,1	26,7	23,0	14,6	20,2	13,8	30	50	0
14:38:17	22,7	23,7	89,1	88,3	87,9	87,2	85,5	83,5	81,3	80,8	80,4	80,3	27,0	23,1	14,7	20,2	14,4	30	50	0
14:38:47	22,6	23,7	89,2	88,3	88,1	87,4	85,5	83,6	81,5	80,9	80,6	80,3	27,0	23,0	14,8	20,2	13,7	30	50	0
14:39:17	22,7	23,6	89,3	88,4	88,1	87,5	85,7	83,6	81,5	80,9	80,6	80,3	27,2	23,1	14,8	20,4	14,2	30	50	0
14:39:47	22,6	23,6	89,3	88,5	88,1	87,5	85,8	84,0	81,4	80,9	80,7	80,4	27,5	23,1	14,7	20,2	14,3	30	50	0
14:40:17	22,6	23,7	89,4	88,0	88,1	87,4	85,8	83,9	81,6	80,9	80,6	80,3	27,5	23,1	14,8	20,2	14,0	30	50	0
14:40:47	22,6	23,7	89,4	88,5	88,3	87,5	85,8	83,9	81,5	81,0	80,7	80,4	27,7	23,1	14,7	20,4	13,9	30	50	0
14:41:17	22,6	23,7	89,5	88,5	88,3	87,6	85,8	83,7	81,5	81,0	80,6	80,4	27,8	23,2	14,8	20,3	14,4	30	50	0
14:41:47	22,7	23,7	89,5	88,5	88,3	87,5	86,0	83,6	81,4	81,0	80,8	80,5	27,8	23,1	14,8	20,5	14,2	30	50	0
14:42:17	22,7	23,7	89,5	88,5	88,3	87,5	85,7	83,8	81,7	80,9	80,5	80,2	27,9	23,2	14,9	20,3	14,5	30	50	0
14:42:47	22,6	23,7	89,6	88,6	88,4	87,5	85,6	83,6	81,5	81,0	80,9	80,5	28,1	23,1	14,6	20,2	14,0	30	50	0
14:43:17	22,8	23,7	89,6	88,5	88,4	87,6	85,9	83,9	81,8	81,0	80,9	80,5	28,0	23,1	14,6	20,2	13,7	30	50	0
14:43:47	22,6	23,7	89,8	88,9	88,5	87,8	86,4	83,9	81,7	81,0	80,7	80,6	28,1	23,1	14,7	20,2	14,2	30	50	0
14:44:17	22,8	23,7	89,7	88,8	88,5	87,8	86,2	84,2	81,7	81,2	80,9	80,6	28,2	23,1	14,6	20,2	14,4	30	50	0
14:44:47	22,7	23,7	89,7	88,8	88,5	87,8	86,3	84,1	81,8	81,2	80,7	80,5	28,0	23,1	14,7	20,2	14,0	30	50	0
14:45:17	22,6	23,8	89,9	88,8	88,5	88,0	86,4	84,2	81,7	81,2	80,7	80,5	28,1	23,1	14,7	20,4	14,4	30	50	0
14:45:47	22,6	23,7	89,9	89,0	88,5	88,0	86,4	84,3	81,8	81,2	80,9	80,6	28,2	23,1	14,7	20,2	14,5	30	50	0
14:46:17	22,7	23,7	89,9	88,9	88,6	88,0	86,5	84,4	82,0	81,3	81,0	80,7	28,3	23,1	14,7	20,2	14,0	30	50	0
14:46:47	22,6	23,7	89,9	89,1	88,8	88,0	86,6	84,4	82,0	81,3	80,9	80,8	28,2	23,1	14,9	20,2	14,6	30	50	0
14:47:17	22,7	23,7	90,0	89,1	88,9	88,3	86,6	84,2	81,4	81,1	80,6	80,6	28,3	23,1	14,7	20,2	14,0	30	50	0

Tab. 6.8 V3, measured values at start of rectification

Temperatures and differential pressure according to flow diagramm / system diagram.
 Y1, Y2, Y3: Controller Outputs for heater, Reflux ratio, frequency of pump drive.

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t hh:mm:ss	T1 °C	T2 °C	T3 °C	T4 °C	T5 °C	T6 °C	T7 °C	T8 °C	T9 °C	T10 °C	T11 °C	T12 °C	T13 °C	T14 °C	T15 °C	T16 °C	P1 mbar	Y1, P _{ei} %	Y2, R %	Y3, f %
15:05:47 22.6	23.8	92.0	91.0	90.8	90.5	88.9	86.5	83.4	82.1	81.7	81.6	28.0	23.2	14.9	20.3	13.8	30	50	0	
15:06:17 22.7	23.8	92.0	91.3	90.9	90.4	88.8	86.6	83.5	82.0	81.9	81.6	28.1	23.1	14.7	20.3	13.4	30	50	0	
15:06:47 22.6	23.9	92.2	91.2	90.9	90.4	88.8	86.6	84.0	82.0	81.7	81.4	28.1	23.2	14.9	20.2	13.1	30	50	0	
15:07:17 22.6	23.9	92.1	90.2	89.3	84.3	82.8	82.6	82.1	81.7	81.4	81.2	28.1	23.2	14.9	17.4	10.1	0	50	0	
15:07:47 22.6	23.8	92.1	83.4	83.3	82.3	81.7	81.8	81.7	81.4	81.1	80.8	27.9	23.1	14.8	16.4	9.2	0	50	0	
15:08:17 22.6	23.7	92.1	82.7	82.5	82.0	81.3	81.6	81.5	81.2	80.5	80.2	27.7	23.2	14.9	16.0	6.8	0	50	0	
15:08:47 22.8	23.9	92.1	82.3	82.2	81.7	81.1	81.2	81.4	80.4	80.3	80.0	27.8	23.1	14.8	15.6	4.9	0	50	0	
15:09:17 22.7	23.8	92.1	82.0	81.8	81.4	80.9	81.4	80.9	80.2	79.8	79.5	27.8	23.2	14.8	15.5	3.8	0	50	0	
15:09:47 22.8	23.8	92.2	81.7	81.5	81.2	80.8	81.1	80.6	79.9	79.5	78.6	27.6	23.2	14.6	15.4	2.7	0	50	0	
15:10:17 22.6	23.8	92.1	81.3	81.5	81.2	80.6	80.6	80.0	79.2	78.4	77.9	27.6	23.2	14.8	15.3	1.8	0	50	0	
15:10:47 22.6	23.8	92.1	81.3	81.2	80.8	80.2	80.3	79.5	78.4	77.9	77.1	27.6	23.3	14.8	15.3	0.1	0	50	0	
15:11:17 22.6	23.7	92.0	81.0	80.8	80.1	79.0	79.0	78.9	78.2	77.9	76.8	27.5	23.2	14.6	15.2	-0.1	0	100	0	
15:11:47 22.7	23.9	92.0	80.3	79.6	79.2	78.1	77.9	77.1	76.5	75.6	75.3	27.2	23.2	14.5	15.3	-0.1	0	100	0	
15:12:17 22.9	24.0	91.9	80.1	79.0	78.4	77.2	77.0	76.3	75.4	74.8	74.5	27.2	23.2	14.6	15.2	-0.1	0	100	0	
15:12:47 22.6	23.9	91.9	79.8	78.1	77.4	76.5	76.2	75.5	74.9	74.2	73.8	27.2	23.2	14.5	15.2	0.1	0	100	0	
15:13:17 22.6	23.9	91.8	79.3	77.1	76.8	75.7	75.5	74.9	74.2	73.4	72.9	27.2	23.3	14.6	15.2	-0.1	0	100	0	
15:13:47 22.6	24.0	91.9	79.3	76.5	76.0	75.1	74.8	74.4	73.5	72.7	72.1	27.2	23.2	14.6	15.2	0.0	0	100	0	
15:14:17 22.6	24.0	91.8	79.3	75.7	75.0	74.2	74.0	73.7	72.7	71.8	71.4	27.2	23.2	14.6	15.2	0.0	0	100	0	
15:14:47 22.6	23.9	91.8	79.2	75.0	74.5	73.4	72.8	71.9	70.0	66.3	70.5	27.4	23.3	14.7	15.3	0.0	0	100	0	
15:15:17 22.6	23.8	91.8	78.8	74.4	73.7	72.5	72.3	71.9	70.6	69.5	69.9	27.2	23.2	14.6	15.2	0.1	0	100	0	
15:15:47 22.6	24.0	91.8	78.5	73.7	73.2	72.0	71.6	71.4	70.1	69.3	69.1	27.2	23.1	14.8	15.3	0.0	0	100	0	
15:16:17 22.6	23.7	91.7	78.5	73.2	72.3	71.1	71.0	70.7	69.4	68.8	68.6	27.2	23.2	14.7	15.3	-0.1	0	100	0	
15:16:47 22.6	24.0	91.7	78.2	72.7	71.6	70.6	70.2	70.2	68.8	68.1	67.7	27.1	23.3	14.9	15.5	-0.1	0	100	0	
15:17:17 22.6	23.8	91.7	78.3	71.8	70.9	69.9	69.7	69.3	68.3	67.5	67.2	27.1	23.2	14.8	15.2	-0.1	0	100	0	
15:17:47 22.7	24.0	91.6	78.1	71.2	70.2	69.4	69.1	68.8	67.6	66.9	66.4	27.0	23.2	14.9	15.5	0.0	0	100	0	
15:18:17 22.6	24.0	91.5	77.7	70.8	69.6	68.6	68.5	67.9	66.9	66.4	65.8	27.0	23.2	14.9	15.5	0.0	0	100	0	
15:18:47 22.6	24.0	91.6	77.6	70.5	69.0	68.0	67.8	67.3	66.2	65.8	65.3	27.0	23.2	14.9	15.5	-0.1	0	100	0	
15:19:17 22.7	23.8	91.4	76.6	70.1	68.2	67.4	67.2	66.7	65.6	65.3	64.6	27.0	23.3	14.9	15.5	-0.1	0	100	0	
15:19:47 22.7	23.9	91.4	77.3	69.6	67.4	66.9	66.5	66.1	65.0	64.7	64.1	27.0	23.2	14.9	15.5	0.0	0	100	0	
15:20:17 22.7	23.9	91.4	77.1	68.8	67.0	66.1	65.9	65.5	64.5	64.2	63.4	26.9	23.3	14.9	15.5	0.1	0	100	0	
15:20:47 22.6	23.9	91.3	76.4	68.7	66.4	65.6	65.4	64.9	63.9	63.7	63.0	26.9	23.2	14.9	15.5	-0.1	0	100	0	
15:21:17 22.7	23.9	91.3	77.0	68.3	65.7	64.8	64.7	64.3	63.3	63.1	62.3	26.9	23.2	14.9	15.5	-0.2	0	100	0	
15:21:47 22.8	23.9	91.2	76.8	67.5	65.3	64.2	64.0	63.5	62.7	62.7	62.0	26.9	23.2	14.9	15.5	0.1	0	100	0	
15:22:17 22.7	23.9	91.1	76.8	67.2	64.6	63.7	63.6	63.1	62.2	62.2	61.3	26.8	23.3	14.9	15.5	0.0	0	100	0	
15:22:47 22.6	23.9	91.0	76.5	66.4	64.0	63.1	63.1	62.5	61.7	61.6	60.9	26.9	23.2	14.9	15.5	-0.1	0	100	0	
15:23:17 22.6	23.9	91.1	76.1	66.3	63.4	62.5	62.4	61.9	61.1	61.2	60.4	26.9	23.2	14.9	15.2	-0.1	0	100	0	
15:23:47 22.6	23.8	91.0	76.4	65.6	62.9	61.9	61.9	61.5	60.6	60.6	60.0	26.7	23.2	14.9	15.5	0.0	0	100	0	
15:24:17 22.7	24.0	91.0	75.2	65.6	62.3	61.3	61.3	60.8	60.1	60.1	59.4	26.8	23.2	15.1	15.6	0.0	0	100	0	
16:30:47 23.3	23.9	58.5	29.2	24.8	26.7	29.4	30.4	30.8	30.9	31.0	30.8	24.5	22.2	22.0	20.5	0.0	100	100	0	

Temperatures and differential pressure according to flow diagram / system diagram.
 Y1, Y2, Y3: Controller Outputs for heater, Reflux ratio, frequency of pump drive.

6.3.4.2 Self-recorded measured values (without PC assistance)

The self-recorded measured values are primarily the masses, densities and temperatures of the various mixtures/products. In the course of the experiment they were entered by hand on the "Batch worksheet" (see Tab. 7.1, page 117).

These measured values are presented, together with the evaluation, in chapter 6.3.5.2, Tab. 6.10, page 81.

6.3.5 Evaluation

6.3.5.1 Presentation of automatically recorded measurement data (experiment aim 2)

The bases of the following diagrams are the measured value tables Tab. 6.7, page 75 to Tab. 6.9 . Please note that the diagrams have differing temperature and pressure ranges.

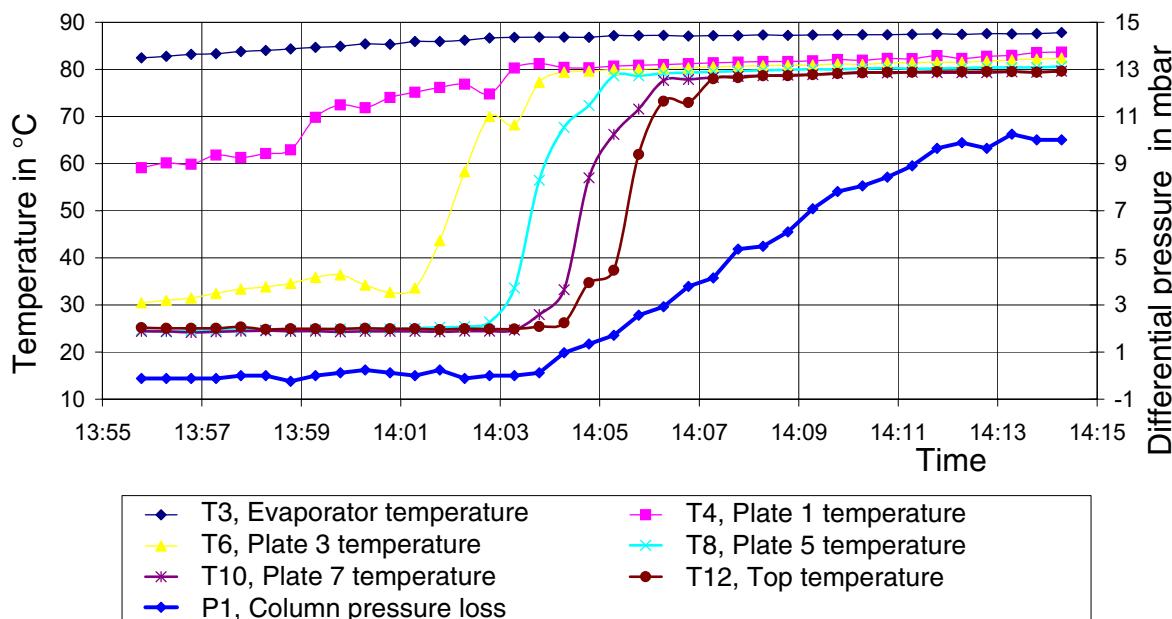


Fig. 6.1 V3, Temperature and pressure profile at column start-up

CE 600

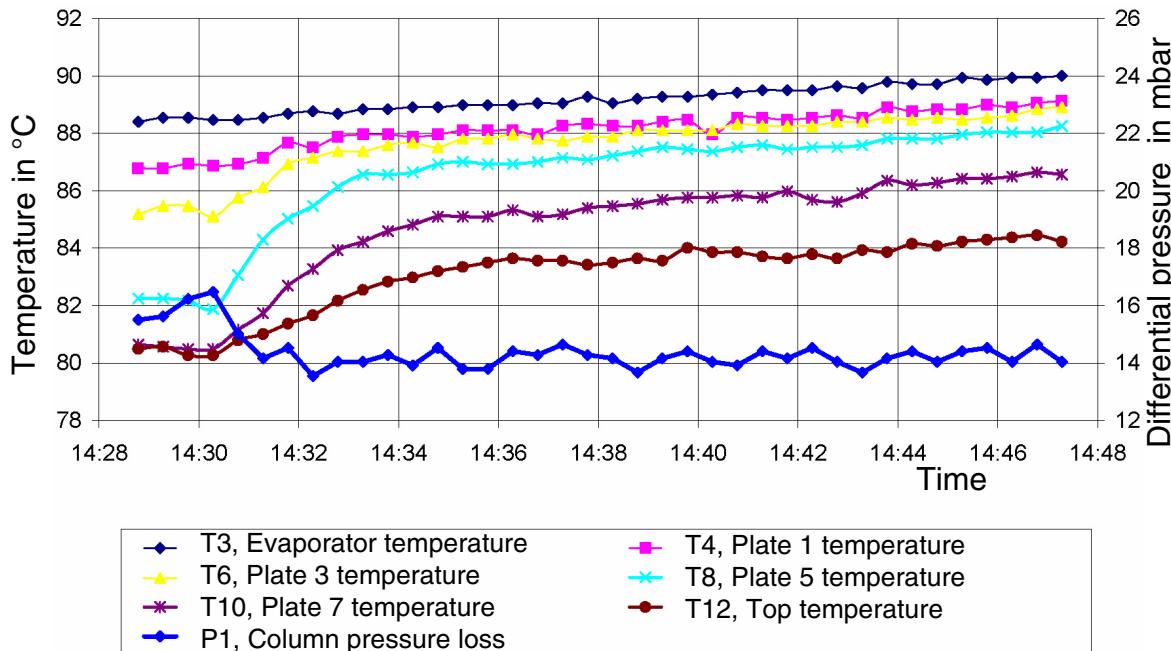
CONTINUOUS RECTIFICATION

Fig. 6.2 V3, Temperature and pressure profile at start of rectification

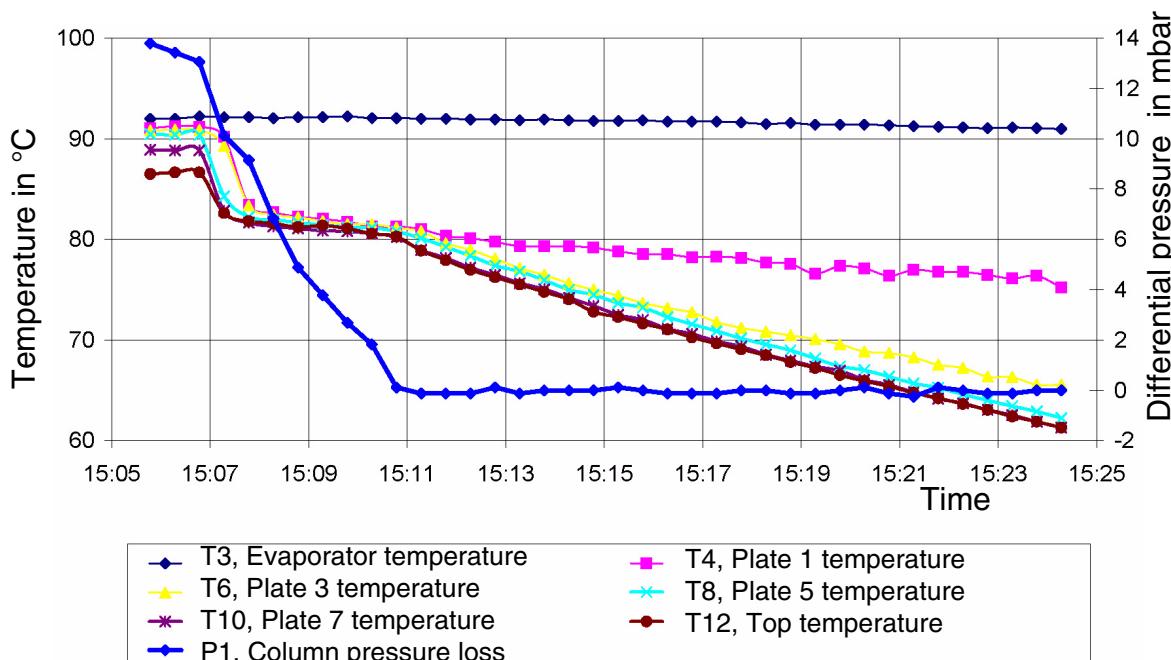


Fig. 6.3 V3, Temperature and pressure profile at end of rectification

6.3.5.2 Evaluation of self-recorded measured values

The following Tab. 6.10, page 81 presents the measured masses, densities and temperatures, together with the total masses and concentrations calculated from them.

The individual mixture masses at the start and end of the experiment have been added together. In determining the total mixture mass at the start of the experiment, the "residue" - here 8,0g - must be taken into account. This "residue" results from the fact that when transferring from the last measuring cup into the feed tank, the total volume of 10,0L is reached before the measuring cup is completely empty. The "residue" is thus the mass of the mixture remaining in the measuring cup.

Re experiment aim 3 - To determine the ethanol concentrations:

For all the samples, the ethanol mass fraction E_{w_E} was determined from the density and temperature by means of linear **interpolation**. The procedure is explained here in respect of the first sample (mixture at start of experiment) by way of example:

The measured values of the first top product sample are:

Total density $\rho_M = 962 \text{ kg/m}^3$ at mixture temperature $T_M = 22,8^\circ\text{C}$ (index M for measured value).

Clock	Product/measurement no.	Measurement					Evaluation	
		Mass net g	Tempera-ture °C	Density kg/L	Level in Evap L	Comments	Mass net g	Mass fraction Ethanol, w_E %m
13:15	Start, mixture, Evap	-----	22,8	0,962	10,0		9695,0	23,8
14:41	T.pr., 1.	430,5	24,0	0,825	9,3		-----	85,9
14:52	T.pr., 2.	370,5	22,7	0,832	9,0		-----	83,6
15:06	T.pr., 3.	429,0	22,8	0,836	9,0		-----	82,0
-----	End, T.pr., mixture	1392,5	22,8	0,832	-----		-----	83,6
-----	End, B.pr., Evap	-----	22,1	0,974	-----		8219,0	15,3

Measurement					
Start, Fill feed tank, Individual masses in g	1945,0	1932,5	1930,5	1942,0	1953,0 (-8,0g residue, when 10L reached)
End, B.pr., from Evap, Individual masses in g	2022,0	2046,0	1904,0	1788,5	458,5

Key: T.pr.: Top product; B.pr.: Bottom product; Evap: Evaporator

Tab. 6.10 V3, Self-recorded measured values, with evaluation

Consequently, w_E cannot be read directly from Tab. 6.1, page 55 because there ρ is listed only for integer w_E and integer T . The four ρ values for measured value 962kg/m^3 belonging to the next highest and next lowest integer values w_E and T can be read however:

$$\rho_A = \rho(23\%\text{m}, 22^\circ\text{C}) = 963,51 \text{ kg/m}^3$$

$$\rho_B = \rho(23\%\text{m}, 23^\circ\text{C}) = 963,01 \text{ kg/m}^3$$

$$\rho_C = \rho(24\%\text{m}, 22^\circ\text{C}) = 962,05 \text{ kg/m}^3$$

$$\rho_D = \rho(24\%\text{m}, 23^\circ\text{C}) = 961,53 \text{ kg/m}^3$$

As a result, $\rho_{23} = \rho(23\%\text{m}, 22,8^\circ\text{C})$ is produced by linear interpolation as:

$$\rho_{23} = \rho_B + \frac{23^\circ\text{C} - T_M}{23^\circ\text{C} - 22^\circ\text{C}} \cdot (\rho_A - \rho_B)$$

$$\rho_{23} = 963,01 + \frac{23^\circ\text{C} - 22,8^\circ\text{C}}{23^\circ\text{C} - 22^\circ\text{C}} \cdot (963,51 - 963,01) = 963,11 \text{ kg/m}^3$$

Correspondingly, for $\rho_{24} = \rho(24\%\text{m}, 22,8^\circ\text{C})$ the value $961,63 \text{ kg/m}^3$ is obtained.

The value w_E being sought is obtained by linear interpolation with the aid of ρ_{23} and ρ_{24} as per:

$$w_E = 23 + \frac{\rho_{23} - \rho_M}{\rho_{23} - \rho_{24}} \cdot (24 - 23)$$

$$w_E = 23 + \frac{963,11 - 962}{963,11 - 961,63} \cdot (24 - 23) = 23,75 \% \text{m}$$

After rounding, this produces the desired ethanol mass fraction of $w_E = 23,8 \% \text{m}$.

Re experiment aim 4. To present the change over time in the ethanol concentration in the top product

Fig. 6.4 shows the progression over time of the ethanol concentration in the top product:

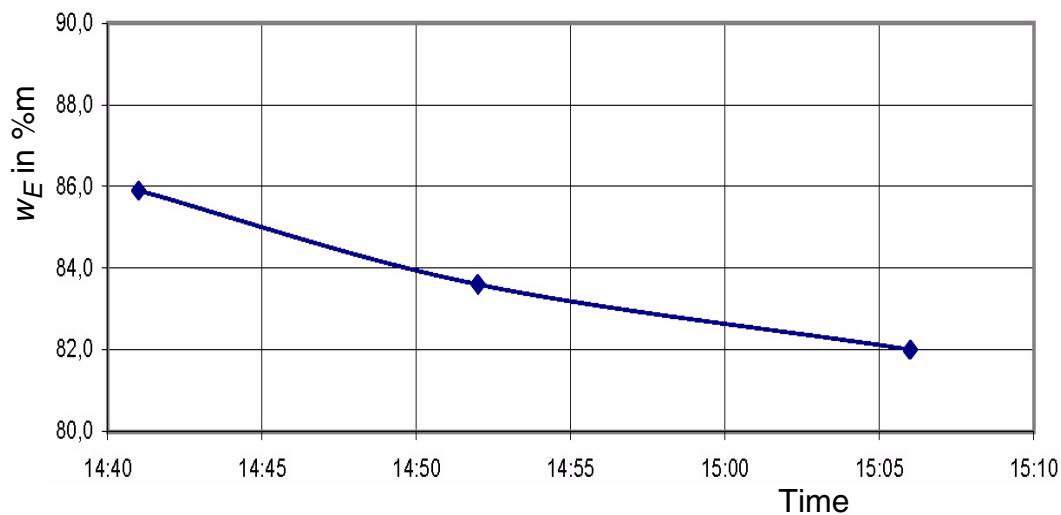


Fig. 6.4 V3, Progression over time of the ethanol concentration in the top product

Re experiment aim 5. To check the concentrations by means of mass balances

It is useful to check the measured values with the aid of mass balances. Any conflicts arising from the mass balances indicate errors in evaluation or inaccurate measurement data.

At this point the mixture and ethanol masses at the start and end of the experiment are compared:

The following examples illustrate the graphical symbols used below:

m :	Mixture mass, general
m_E :	Ethanol mass, general
w_E :	Ethanol mass fraction, general
$m(Start)$:	Mixture mass, at start of experiment
$m_E(End)$:	Ethanol mass, at end of experiment
$w_E(T.pr.)$:	Ethanol mass fraction in top product
$m(T.pr.)$:	Mixture mass, top product

Mixtures mass balance:

To be checked:

$$m(Start) = m(End) \quad (6.1)$$

where

$$m(End) = m(T.pr.) + m(B.pr.,Evap) \quad (6.2)$$

The following table consolidates the values from Tab. 6.10, page 81 and adds them together according to the formula (6.2):

	Mass, net, in g	
	<i>m(Start)</i>	<i>m(End)</i>
<i>m(Start)</i>	9695,0	-----
<i>m(T.pr.)</i>	-----	1392,5
<i>m(B.pr.,Evap)</i>	-----	8219,0
Total	9695,0	9611,5

Tab. 6.11 V3, Mixture balancing

The comparison of the two values shows that at the end of the experiment there is still **99,1%** of the original mixture mass present.

Ethanol mass balance:

In line with the mass balances for the mixtures, (6.1) and (6.2), the resultant ethanol mass balances are:

To be checked:

$$m_E(\text{Start}) = m_E(\text{End}) \quad (6.3)$$

where

$$m_E(\text{End}) = m_E(\text{T.pr.}) + m_E(\text{B.pr.,Evap}) \quad (6.4)$$

The ethanol mass fraction w_E is defined as the ratio of the ethanol mass m_E to the mixture mass m . This enables the individual ethanol masses to be expressed as the product of the mixture mass and the mass fraction, e.g. for:

$$m_E(\text{T.pr.}) = m(\text{T.pr.}) \times w_E(\text{T.pr.}) \quad (6.5)$$

The following table summarises how the values from Tab. 6.10, page 81 and Tab. 6.11, page 85 are evaluated using the equations (6.4) and (6.5):

Designation ...	Start			End		
	$m(\dots)$	$w_E(\dots)$	$m_E(\dots)$	$m(\dots)$	$w_E(\dots)$	$m_E(\dots)$
	g	%m	g	g	%m	g
Start	9695,0	23,8	2307,5	-----	-----	-----
T.pr.	-----	-----	-----	1392,5	83,6	1164,0
B.pr.,Evap	-----	-----	-----	8219,0	15,3	1257,5
Total	9695,0	-----	2307,5	9611,5	-----	2421,5

Tab. 6.12 V3, Ethanol balancing

The comparison of the two values shows that at the end of the experiment there is still **104,9%** of the original ethanol mass present.

This surprising result is commented upon in chapter 6.3.6 under item 5.

6.3.6 Comments, assessment

The comments and assessment are based on the pre-defined experiment aims:

- Re 1. To concentrate the ethanol and
Re 3. To determine the ethanol concentration
in the initial mixture, top product and bottom
product**

The initial mixture was separated into the top product, with increased ethanol content, and the bottom product, with reduced ethanol content.

The ethanol mass fraction in the top product is greater than in the initial mixture by a factor of **3,51**.

- Re 2. To record the measured value using the
data acquisition program (temperature and
pressure profiles)**

Fig. 6.1, page 78 shows the profiles at column start-up. The temperature rise in the individual plates from bottom to top is clearly traceable. Some of the temperature curves have a kink in them during heat-up. A kink of this kind can be interpreted as forming the equilibrium between vapour and liquid, on occurrence of the first condensate.

While the equilibria are being created, the pressure loss in the column increases steadily.

The time between reaching the boiling temperature in the column head and the initial distillate in the phase separation tank (IV) is about 5 minutes under the selected conditions. When the first distillate occurs, the equilibria have formed and the differential pressure in the column increases much less markedly.

Fig. 6.2, page 79 shows the differential pressure and temperatures at the start of rectification. At 14:30 hours the operating conditions are adjusted (reflux ratio R from 100 to 50%, heater from 20 to 30%). The effect of the increase in heating power is felt only slowly, identifiable by the flat rate of rise of the curve for the evaporator temperature T3.

A rapid reaction does occur, however, because of the reduced reflux. The temperatures on the upper plates 5 and 7, in particular, rise rapidly. The cause of this is the halving of the much cooler reflux flow into the column head (reflux temperature T13 at 14:30 around 25°C). That is to say, the mixture of cooler reflux and newly formed condensate on the plates becomes warmer. New equilibriums are formed.

Fig. 6.3, page 79 shows the profiles at the end of rectification. Operations are aborted by switching off the heater (0%) at around 15:06 hours. The reaction of the temperatures in the column and column head is rapid. An initial rapid drop in temperatures by 4 to 8°C is detected. The fastest reaction is on the column head and the upper plates. At the same time the steam pressure falls, so the column loses condensate.

The reflux ratio R was changed from 50 to 100% at 15:11 hours. The purpose of this delay 5 minutes after the actual abort is to obtain as much top product as possible under conditions similar to those during operation.

Then the last top product sample is taken.

As a reaction to the larger flow of cooler reflux (at 15:11 hours around 27°C) into the column head, an accelerated fall in the temperatures in the upper area of the column is detected.

Re 4. To present the change over time in the ethanol concentration in the top product

Fig. 6.4, page 83 shows the decrease in the ethanol mass fraction w_E in the top product. It is seen that w_E decreases by 3,9 percentage points in the course of the experiment.

At first glance that appears a substantial decrease.

The reason for this decrease is that there is a greater proportion of ethanol in the newly created top product than in the bottom product in the evaporator. As a result, the bottom product in the evaporator loses ethanol in the course of the experiment. The decreasing concentration in the evaporator then results in a reduced concentration in the top product.

Re 5. To check the concentrations by means of mass balances

Evaluation of the experiment shows that at the end of the experiment there is still **99,1%** of the original mixture mass present.

Taking into account the losses during sampling and transfer, and due to evaporation, these values are of a realistic magnitude.

By contrast, it is surprising that at the end of the experiment there is still **104,9%** of the original ethanol mass present.

There cannot be an actual increase in ethanol mass, because no chemical conversion takes place in the rectification unit. A similar loss as in the case of the mixture masses would be expected.

Consequently, an investigation is conducted as to whether a realistic value is produced when measurement inaccuracies are taken into account.

Of importance to the balancing operation are masses and mass fractions. The accuracy in determining masses is extremely high.

The influence of the mass fractions on the balancing result is much more marked, because both the temperature and the density are incorporated into the calculation of mass fractions.

Of these two measured values, the density has the greater influence.

To measure the density, the areometer shown adjacent was used. The measurement error for this areometer is composed of the actual instrument error



Fig. 6.5 Areometer used

(Scale does not precisely match the actual penetration depth) and the read error (meniscus formation on the scale, parallax).

The measurement error for the areometer used is unfortunately not specified. Consequently, only the effect of the read error is investigated here:

The measuring range of the areometer is 0,800 to 1,000kg/L. The difference in density between two graduations on the scale is 0,002kg/L. Consequently, the read error is estimated as $\pm 0,001\text{ kg/L}$.

The following Tab. 6.13 shows how the variation in density by 0,001kg/L affects the ethanol mass fraction w_E (corrected values are shown here in blue).

Designation ...	Values		Corrected values	
	$\rho(\dots)$ kg/L	$w_E(\dots)$ %m	$\rho_K(\dots)$ kg/L	$w_{E,K}(\dots)$ %m
Start	0,962	23,8	0,961	24,4
T.pr.	0,832	83,6	0,833	83,2
B.pr.,Evap	0,974	15,3	0,975	14,6

Tab. 6.13 V3, Influence of density measurement error on ethanol mass fraction

These corrected values result in the following balance (see Tab. 6.14):

Designation ...	Start			End		
	$m(\dots)$	$w_{E,K}(\dots)$	$m_{E,K}(\dots)$	$m(\dots)$	$w_{E,K}(\dots)$	$m_{E,K}(\dots)$
<i>Start</i>	9695,0	24,4	2366,0	-----	-----	-----
<i>T.pr.</i>	-----	-----	-----	1392,5	83,2	1158,5
<i>B.pr.,Evap</i>	-----	-----	-----	8219,0	14,6	1200,0
Total	9695,0	-----	2366,0	9611,5	-----	2358,5

Tab. 6.14 V3, Ethanol balancing, with influence of density measurement error

The comparison of the two values shows that at the end of the experiment there is now still **99,7%** of the original ethanol mass present.

The apparent contradiction can thus be explained solely by the areometer read error.

The influence of the densities here is so great that a change in density of 0,001 kg/L alters the balance by more than 5 percentage points.

6.4 Example of a continuous experiment, V7

6.4.1 Experiment aims

Experiment aims are:

1. To concentrate the ethanol
2. To record the measured value using the data acquisition program (temperature and pressure profiles)
3. To determine the ethanol concentration in the initial mixture, top product and bottom product
4. To present the change over time in the ethanol concentration in the top and bottom products
5. To check the concentrations by means of mass balances

6.4.2 Experimental setup

Connected CE 600 unit, with accessories as set out in chapter 5.2, page 37.

6.4.3 Performing the experiment

- Observe the safety instructions (see chapter 3, page 19)
- Prepare the unit as set out in chapter 5.5.1, page 44. Open the manual stop valve underneath the pressure gauge.
- The requirement is a canister containing more than 19L of initial mixture, ethanol: 25%m, water: 75%m (new addition, e.g. for 20L, with 4808g ethanol and 14425g water).
- Mix the initial mixture in the canister.

- Identify the density and temperature of the initial mixture. Check the concentration.
- Transfer the initial mixture from the canister into the two feed tanks (VI). Use measuring cups for this, and weigh the content of each cup (net weight of each). Fill the feed tanks up to their 5 litre marks. Also weigh the residue in the measuring cup (after pumping out, pour into feed tank).
- Transfer 9 of the 10L of initial mixture from the feed tanks through the column into the evaporator (see chapter 5.6.2, page 47). While transferring, open the stop valve on the column. Switch the pump off when the 9L level is reached according to the markings on the feed tanks.
- Transfer additional initial mixture from the canister into the two feed tanks. Use measuring cups for this, and weigh the content of each cup (net weight of each). Fill the feed tanks up to their 5L marks. Also weigh the residue in the measuring cup.
- Check whether the heat exchanger (VII) is switched for feed heating.
- Press Reset on the differential pressure display (item 23 in Fig. 2.6, page 10).
- Unit operation by PC. Make the settings for data acquisition, open the measured value file (see chapter 2.4.2, page 13).
- Set the reflux ratio to 100% initially
- Open the condenser water supply and adjust it to about 300 L/h.
- Switch on the heater. Initial fixed value 100%.

- When evaporator temperature $T_3 = 80^\circ\text{C}$ is reached, reduce the heater to the fixed value 25%.
- Wait until distillate appears at the top tube of the phase separation tank (IV) and reflux begins. After waiting 5 minutes, change operating conditions:
 - Start feed in column, pump (X) on at 20%
 - Reflux ratio from 100 to 25%
 - Open valve slightly from evaporator to front bottom product tank (see chapter 5.5.1, page 44).
 - Note down the volume in the evaporator
 - Keep the level in the evaporator as constant as possible, adjusting the valve setting as necessary.
- When the minimum sample volume is reached, withdraw the top product from the top product tank (V). Determine the mass, density and temperature. Keep the sample (for a later mixed sample). Note down the volume in the evaporator. Perform a new measurement as soon as the minimum sample volume has been reached again.
- When the minimum sample volume is reached, withdraw the bottom product from the bottom product tank (VIII). Determine the mass, density and temperature.
Keep the sample (for a later mixed sample). Note down the volume in the evaporator. Perform a new measurement as soon as the minimum sample volume has been reached again.

- Abort e.g. after adding 5L feed:
 - Pump off
 - Heater off
 - Close valve from evaporator to bottom product tank
- 5 minutes after aborting, change reflux ratio to 100%. Shut off the water supply.
- Measure the remaining bottom product (totally drain bottom product tank); determine mass, density and temperature.
- Pour together and intermingle the previous bottom product samples and measure the total mass, density and temperature.
- Measure the remaining top product (residue from top product tank together with residue from phase separation tank); determine the mass, density and temperature.
- Pour together and intermingle the previous top product samples and measure the total mass, density and temperature.
- Cool the bottom product from the evaporator (see chapter 5.6.3, page 48). Drain off the cooled bottom product, weigh it and mix it in the second canister. Take a sample of the bottom product mixture, determine density and temperature.
- Fill canister with unused initial mixture and with top product and the two bottom products. Mix well.
This provides a new initial mixture for the next experiment.

6.4.4 Measured values

6.4.4.1 Measurement data from the data acquisition program (experiment aim 2)

Using the data acquisition program, the measured values were saved in “.dat” file format. This **measured value file** contains a chronological sequence of measured data records. A **measured data record** is a snapshot of all the measured values at the given point in time (see also chapter 2.4.2, page 13).

For this experiment an interval of 30 seconds was selected (representing the time between recording of two measured data records). Based on a minimum recording period of 120 minutes, this produces more than 5040 measured values (two measured data records per minute, each with 21 measured values).

A tabular representation of the complete measured value file, with all measured data records, would be too extensive to set out at this point. Consequently, a selection was made to depict the most informative time bands.

The selected time bands are identical to the batch experiment:

- Column start-up until production of the first distillate
- Start of rectification under operating conditions
- End of rectification under operating conditions

The measured values for the selected time bands are imported into MS-Excel (see measured value tables Tab. 6.15 to Tab. 6.17). The MS-Excel Import function is used to import the measured values.

Alongside the differential pressure P1 and the temperatures T1 to T16, these measured value tables also contain the three control variables Y1 to Y3. In the experiment described here, fixed values were programmed on PC rather than using the software controllers. Using the defined operating conditions (heater $Y1=25\%$, reflux ratio $Y2=25\%$, pump $Y3=20\%$), this enables the start and end of the actual experiment (here 16:35 and 17:44 hours) to be determined from the measured value tables.

The graphical view is presented in chapter 6.4.5.1, page 102.

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t hh:mm:ss	T1 °C	T2 °C	T3 °C	T4 °C	T5 °C	T6 °C	T7 °C	T8 °C	T9 °C	T10 °C	T11 °C	T12 °C	T13 °C	T14 °C	T15 °C	T16 °C	P1 mbar	Y1, P _e %	Y2, R %	Y3, f %	
15:59:36	24,0	24,6	26,7	25,1	25,0	24,2	23,6	24,2	24,2	24,0	24,1	23,7	22,9	23,2	23,8	0,0	0	100	0		
16:10:36	22,9	24,0	82,2	52,9	27,5	24,2	23,7	24,0	24,1	24,0	24,1	23,8	23,1	23,5	24,0	-0,1	25	100	0		
16:11:36	22,9	24,0	82,6	53,7	27,8	24,1	23,6	24,0	24,0	24,0	24,0	23,9	22,9	22,3	22,8	0,4	25	100	0		
16:12:06	22,9	24,0	82,8	54,9	28,0	24,0	23,7	24,0	24,1	24,0	24,0	23,7	22,8	22,5	16,5	16,8	0,0	25	100	0	
16:12:36	22,9	24,0	83,2	53,8	28,3	24,1	23,7	24,1	24,0	24,0	23,8	24,0	23,7	22,9	14,4	14,9	0,1	25	100	0	
16:13:06	22,8	24,1	83,6	55,3	28,9	24,3	23,7	24,2	24,1	24,0	24,0	23,9	22,9	14,1	14,6	0,0	25	100	0		
16:13:36	22,9	24,0	84,2	58,1	30,3	24,2	23,7	24,0	24,0	24,1	24,0	23,8	23,9	22,9	13,9	14,3	0,0	25	100	0	
16:14:06	22,6	23,8	85,0	61,1	30,8	24,2	23,7	24,0	24,0	24,2	24,0	24,1	23,9	22,9	13,8	14,3	0,1	25	100	0	
16:14:36	22,6	24,0	85,3	60,0	31,6	24,3	23,7	23,9	24,1	24,1	24,0	24,0	23,7	23,0	13,8	14,3	0,0	25	100	0	
16:15:06	22,7	23,8	85,5	63,6	32,3	24,3	23,7	24,0	24,0	24,0	24,0	24,0	24,0	23,7	23,0	14,1	14,5	0,1	25	100	0
16:15:36	22,6	23,9	86,0	67,8	33,7	24,5	23,7	23,9	24,1	24,0	24,1	24,0	23,7	22,9	13,9	14,3	0,0	25	100	0	
16:16:06	22,6	23,7	86,4	68,0	38,7	24,5	23,7	24,0	24,0	24,0	24,2	24,2	23,9	22,9	13,8	14,1	0,1	25	100	0	
16:16:36	22,6	23,9	86,6	70,8	52,7	24,8	23,7	23,9	24,0	24,0	23,9	23,9	24,0	22,9	13,8	14,1	0,0	25	100	0	
16:17:06	22,6	24,0	87,0	72,3	64,7	27,3	23,7	23,9	24,0	24,1	24,1	24,0	24,0	23,1	13,8	14,1	0,1	25	100	0	
16:17:36	22,8	24,0	87,2	80,0	69,1	36,3	24,1	24,1	24,1	24,0	24,0	24,0	24,1	23,8	23,0	13,8	14,1	0,4	25	100	0
16:18:06	22,6	23,9	87,4	81,4	79,3	61,4	28,3	24,8	24,0	24,0	23,9	23,9	23,0	13,8	14,1	1,5	25	100	0		
16:18:36	22,7	23,8	87,4	81,2	79,8	71,1	49,5	28,3	24,1	24,0	24,0	24,1	23,7	22,9	13,8	14,1	1,8	25	100	0	
16:19:06	22,6	23,8	87,5	81,7	80,6	78,7	68,8	33,1	24,1	24,2	24,0	24,0	23,9	23,0	13,8	14,2	2,9	25	100	0	
16:19:36	22,6	23,9	87,7	82,2	80,6	79,5	77,0	65,0	24,2	24,1	24,0	24,0	23,9	23,0	13,6	14,1	4,0	25	100	0	
16:20:06	22,6	23,9	87,7	82,5	80,9	79,8	78,4	72,7	35,4	24,0	24,0	24,0	23,7	23,0	13,8	14,2	4,9	25	100	0	
16:20:36	22,6	23,9	87,5	82,8	80,9	79,9	79,2	78,5	71,2	24,1	24,0	23,9	23,9	22,9	13,7	14,1	6,0	25	100	0	
16:21:06	22,6	23,8	87,9	83,4	81,4	80,3	79,3	79,2	78,1	55,4	24,0	24,0	23,7	23,1	13,8	14,1	6,6	25	100	0	
16:21:36	22,6	23,7	87,8	83,8	81,8	80,5	79,4	79,2	79,0	73,2	25,8	24,0	23,8	22,9	13,8	14,2	7,1	25	100	0	
16:22:06	22,6	23,7	88,0	83,8	82,3	80,6	79,5	79,5	78,4	51,0	24,2	23,9	23,1	23,8	14,1	8,4	25	100	0		
16:22:36	22,6	24,0	88,0	84,5	82,6	80,9	79,8	79,7	79,5	79,1	73,2	63,4	23,8	23,0	13,8	14,1	9,9	25	100	0	
16:23:06	22,6	23,9	87,9	84,8	83,1	81,3	79,9	80,1	79,7	79,2	78,7	74,1	23,7	23,1	13,8	14,1	10,4	25	100	0	
16:23:36	22,5	23,7	88,2	85,3	83,7	81,5	80,1	80,1	79,8	79,4	78,7	78,7	23,7	23,1	13,8	14,1	11,0	25	100	0	
16:24:06	22,6	23,7	88,2	85,2	84,1	81,8	80,4	80,1	79,9	79,4	79,0	23,8	23,1	13,8	14,2	11,4	25	100	0		
16:24:36	22,6	23,7	88,1	85,9	84,6	82,0	80,4	80,3	79,8	79,5	79,2	23,7	23,1	13,8	14,1	12,0	25	100	0		
16:25:06	22,6	23,8	88,3	86,4	85,0	82,7	80,8	80,3	79,9	79,6	79,3	23,8	23,1	13,7	15,2	12,2	25	100	0		
16:25:36	22,5	23,7	88,3	86,5	85,2	83,1	80,9	80,4	80,1	79,8	79,5	23,7	22,9	13,8	15,5	12,1	25	100	0		
16:26:06	22,5	23,8	88,2	86,6	85,3	83,5	81,2	80,6	80,2	79,8	79,5	23,7	22,9	13,7	15,7	12,3	25	100	0		
16:26:36	22,7	23,6	88,5	86,6	85,5	83,8	81,4	80,8	80,3	79,8	79,7	23,8	23,1	13,8	15,7	12,0	25	100	0		
16:27:06	22,6	23,9	88,3	86,6	86,0	84,0	81,8	81,0	80,4	80,0	79,8	79,6	23,8	23,0	13,8	15,7	11,8	25	100	0	
16:27:36	22,6	23,8	88,4	87,0	86,2	84,4	82,0	81,1	80,5	80,2	80,0	79,7	23,9	22,9	13,8	15,8	12,0	25	100	0	
16:28:06	22,6	23,9	88,4	87,1	86,4	84,8	82,3	81,2	80,7	80,1	80,0	79,6	23,7	23,1	13,8	16,0	11,6	25	100	0	
16:28:36	22,4	23,7	88,3	87,2	86,6	85,0	82,6	81,3	80,6	80,3	80,1	79,7	23,7	22,8	13,8	16,1	11,6	25	100	0	
16:29:06	22,5	23,7	88,5	87,3	86,6	85,1	82,8	81,3	80,6	80,2	80,1	80,0	23,9	23,0	13,8	16,0	11,4	25	100	0	
16:29:36	22,7	23,8	88,5	87,5	86,6	85,3	83,1	81,6	80,9	80,2	80,1	79,9	24,0	23,1	13,8	16,0	11,0	25	100	0	

Temperatures and differential pressure according to flow diagramm / system diagram.
 Y1, Y2, Y3: Controller Outputs for heater, Reflux ratio, frequency of pump drive.

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t hh:mm:ss	T1 °C	T2 °C	T3 °C	T4 °C	T5 °C	T6 °C	T7 °C	T8 °C	T9 °C	T10 °C	T11 °C	T12 °C	T13 °C	T14 °C	T15 °C	T16 °C	P1 mbar	Y1, P _{el} %	Y2, R %	Y3, f %
16:34:36	22,6	23,6	89,1	87,9	87,4	85,9	83,5	81,7	80,7	80,3	80,1	79,8	25,6	23,1	14,0	16,0	14,9	25	100	0
16:35:06	22,6	23,8	89,0	87,7	87,4	86,1	83,6	81,7	80,6	80,3	80,0	79,7	25,8	23,1	14,0	16,0	15,1	25	100	0
16:35:36	24,3	23,2	88,9	87,2	86,1	85,8	83,3	81,6	80,7	80,3	80,1	79,7	25,3	23,0	14,1	15,7	16,6	25	25	20
16:36:06	24,2	22,9	89,1	86,6	85,7	84,2	81,6	80,7	80,6	80,3	80,0	79,8	25,3	22,9	14,0	15,5	17,6	25	25	20
16:36:36	24,1	27,6	89,2	87,1	86,7	82,5	80,6	80,5	80,6	80,3	80,1	79,9	25,5	23,9	14,1	15,5	17,6	25	25	20
16:37:06	24,2	30,3	89,1	87,5	86,6	81,7	80,3	80,3	80,5	80,2	80,1	79,8	25,3	23,5	13,9	15,4	17,6	25	25	20
16:37:36	24,0	28,6	89,3	87,5	86,7	81,4	80,1	80,4	80,4	80,1	80,0	79,9	25,3	23,7	14,0	15,3	18,3	25	25	20
16:38:06	24,2	27,0	89,2	87,4	86,6	81,7	80,2	80,5	80,5	80,2	80,0	79,8	25,3	23,7	14,0	15,4	18,7	25	25	20
16:38:36	24,2	25,9	89,3	87,2	86,6	81,9	80,5	80,6	80,6	80,5	80,1	79,8	25,3	23,9	13,9	15,5	18,1	25	25	20
16:39:06	24,3	25,6	89,3	87,2	86,3	82,0	80,6	80,5	80,7	80,3	80,1	79,9	25,3	23,8	13,9	15,5	17,3	25	25	20
16:39:36	24,4	25,4	89,4	87,2	86,0	82,0	80,6	80,7	80,6	80,4	80,2	80,0	25,1	23,7	13,8	15,5	16,7	25	25	20
16:40:06	24,3	25,4	89,4	87,0	85,8	81,9	80,6	80,6	80,6	80,3	80,1	80,1	25,0	23,9	13,8	15,3	16,4	25	25	20
16:40:36	24,3	25,4	89,4	86,8	85,5	81,8	80,5	80,6	80,6	80,5	80,3	80,1	25,1	24,0	13,9	15,5	16,1	25	25	20
16:41:06	24,4	25,5	89,4	86,4	85,3	81,8	80,4	80,6	80,6	80,5	80,1	80,1	25,0	24,2	13,8	15,5	16,1	25	25	20
16:41:36	24,3	25,9	89,4	86,4	85,3	81,8	80,6	80,6	80,6	80,5	80,3	79,9	25,1	24,4	14,0	15,4	15,6	25	25	20
16:42:06	24,4	26,4	89,4	86,1	85,0	81,8	80,4	80,8	80,7	80,5	80,3	80,1	25,0	24,2	14,1	15,5	15,6	25	25	20
16:42:36	24,4	27,0	89,4	86,1	85,0	81,6	80,3	80,6	80,6	80,4	80,3	80,2	24,9	24,5	14,1	15,4	15,4	25	25	20
16:43:06	24,2	27,9	89,5	86,1	85,0	81,7	80,4	80,9	80,6	80,4	80,3	80,1	25,0	24,8	14,0	15,5	15,3	25	25	20
16:43:36	24,4	29,4	89,5	86,1	85,0	81,7	80,5	80,6	80,6	80,5	80,3	80,1	25,1	24,8	14,0	15,5	15,1	25	25	20
16:44:06	24,3	31,0	89,5	86,1	85,0	81,8	80,4	80,8	80,7	80,6	80,4	80,2	25,1	24,8	13,8	15,5	15,0	25	25	20
16:44:36	24,2	32,9	89,5	86,1	85,0	81,7	80,5	80,7	80,8	80,5	80,3	80,2	25,0	25,0	14,0	15,5	14,9	25	25	20
16:45:06	24,3	34,6	89,6	86,4	85,0	81,7	80,6	80,6	80,7	80,4	80,3	80,1	25,0	25,2	13,9	15,3	14,8	25	25	20
16:45:36	24,4	36,5	89,5	86,3	85,1	81,9	80,8	81,1	80,8	80,5	80,3	80,3	25,2	25,3	13,8	15,5	14,5	25	25	20
16:46:06	24,4	37,4	89,4	86,4	85,5	82,1	80,7	80,7	80,8	80,5	80,4	80,3	25,0	25,5	13,8	15,4	14,6	25	25	20
16:46:36	24,4	38,5	89,4	86,4	85,7	82,1	80,7	80,8	80,9	80,6	80,3	80,2	25,2	25,6	13,9	15,3	14,0	25	25	20
16:47:06	24,5	39,7	89,6	86,5	85,5	82,3	80,8	80,9	80,9	80,8	80,6	80,4	25,2	25,7	14,0	15,5	14,4	25	25	20
16:47:36	24,5	40,5	89,5	86,4	85,7	82,3	80,9	80,9	80,9	80,6	80,4	80,3	25,3	25,6	14,0	15,5	14,2	25	25	20
16:48:06	24,5	41,5	89,5	86,6	85,6	82,5	80,9	81,0	80,9	80,7	80,5	80,3	25,3	26,0	13,8	15,5	14,0	25	25	20
16:48:36	24,5	42,2	89,6	86,6	85,6	82,6	80,9	81,0	80,9	80,6	80,3	80,3	25,3	25,9	13,8	15,5	14,3	25	25	20
16:49:06	24,5	42,9	89,7	86,5	85,8	82,8	80,9	81,0	80,8	80,6	80,4	80,3	25,3	25,8	14,0	15,5	13,9	25	25	20
16:49:36	24,5	43,7	89,5	86,6	85,8	82,8	80,9	81,0	80,9	80,6	80,4	80,3	25,2	25,9	13,9	15,7	13,8	25	25	20
16:50:06	24,5	44,7	89,7	86,6	85,8	83,0	81,1	80,9	81,0	80,6	80,3	80,3	25,1	26,0	14,0	15,4	13,7	25	25	20
16:50:36	24,5	45,1	89,7	86,7	85,8	83,3	81,2	81,0	80,8	80,6	80,4	80,3	25,3	26,0	14,0	15,5	13,7	25	25	20
16:51:06	24,5	45,9	89,7	86,7	85,6	83,3	81,1	80,9	80,9	80,7	80,5	80,3	25,2	26,1	13,8	15,6	13,3	25	25	20
16:51:36	24,4	46,4	89,6	86,8	85,8	83,5	81,2	81,2	80,9	80,7	80,5	80,3	25,1	26,6	13,9	15,7	12,6	25	25	20
16:52:06	24,3	47,1	89,8	86,9	85,8	83,6	81,4	80,9	80,9	80,6	80,5	80,1	25,4	26,5	14,0	15,5	13,4	25	25	20
16:52:36	24,4	48,0	89,8	86,7	86,0	83,7	81,4	81,2	81,1	80,7	80,6	80,4	25,7	27,5	13,8	15,5	13,1	25	25	20
16:53:06	24,4	49,2	89,7	86,9	86,2	83,9	81,6	81,6	81,1	80,7	80,5	80,3	25,7	28,1	13,8	15,7	13,2	25	25	20
16:53:36	24,4	50,8	89,8	86,9	86,2	83,8	81,5	81,3	81,0	80,7	80,5	80,3	25,8	28,2	13,8	15,4	13,2	25	25	20

Temperatures and differential pressure according to flow diagram / system diagram.
 Y₁, Y₂, Y₃: Controller Outputs for heater, Reflux ratio, frequency of pump drive.

Tab. 6.16 V7, measured values at start of rectification

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CONTINUOUS RECTIFICATION

t hh:mm:ss	T1 °C	T2 °C	T3 °C	T4 °C	T5 °C	T6 °C	T7 °C	T8 °C	T9 °C	T10 °C	T11 °C	T12 °C	T13 °C	T14 °C	T15 °C	P1 mbar	Y1, P _{el} %	Y2, R	Y3, f %	
17:40:36	24.4	63.1	92.3	88.2	86.8	88.2	85.2	85.0	84.2	82.7	82.3	82.3	82.3	82.3	27.5	32.2	14.0	16.0	10.6	25
17:41:06	24.4	63.0	92.3	88.3	86.7	88.2	85.2	84.9	84.3	82.6	82.3	82.2	82.2	82.2	27.4	32.2	13.8	15.9	10.6	25
17:41:36	24.4	63.1	92.4	88.2	86.6	88.2	85.3	85.0	84.3	82.8	82.4	82.2	82.2	82.2	27.2	32.2	13.8	16.0	10.7	25
17:42:06	24.5	63.5	92.4	88.2	86.6	88.2	85.3	85.0	84.2	82.7	82.3	82.3	82.3	82.3	26.9	32.0	13.9	15.9	10.6	25
17:42:36	24.4	63.4	92.4	88.2	86.6	88.1	85.1	85.0	84.2	82.6	82.3	82.3	82.3	82.3	27.0	32.1	13.9	16.0	10.4	25
17:43:06	24.5	63.4	92.4	88.2	86.6	88.1	85.1	85.0	84.3	82.6	82.3	82.1	82.1	82.1	27.1	32.3	14.0	15.9	10.7	25
17:43:36	24.4	63.4	92.4	88.0	86.8	88.1	85.3	85.1	84.2	82.8	82.4	82.1	82.1	82.1	27.3	32.4	13.8	16.0	10.4	25
17:44:06	24.3	63.1	92.2	87.2	86.6	85.6	84.6	84.4	83.1	82.5	82.3	82.0	82.0	82.0	26.2	32.0	14.1	14.8	6.7	0
17:44:36	24.4	62.1	92.4	84.5	84.1	83.4	82.5	82.5	82.5	82.3	81.2	81.0	81.0	81.0	26.9	31.4	14.0	14.5	4.4	0
17:45:06	24.2	61.4	92.4	83.0	82.8	82.6	82.0	82.5	81.3	80.9	80.8	80.7	80.7	80.7	27.0	31.0	13.9	14.5	3.5	0
17:45:36	24.3	60.4	92.4	82.5	82.6	82.3	81.7	82.3	81.7	80.3	80.3	80.1	80.1	80.1	26.7	30.6	13.9	14.6	2.3	0
17:46:06	24.2	59.6	92.4	82.3	82.3	82.1	81.7	81.9	81.9	80.3	80.0	79.6	79.6	79.6	27.0	30.3	13.8	14.4	0.5	0
17:46:36	24.2	58.7	92.4	82.1	82.1	81.7	80.9	80.9	80.0	79.2	78.8	78.5	78.5	78.5	27.2	30.1	14.0	14.4	0.4	0
17:47:06	24.2	57.9	92.4	81.9	81.4	81.0	79.9	79.8	79.1	78.2	77.8	77.6	77.6	77.6	27.2	30.0	13.9	14.5	0.2	0
17:47:36	24.3	57.2	92.4	81.2	80.6	80.1	79.1	78.8	78.1	77.5	76.8	76.7	76.7	76.7	27.4	29.7	13.9	14.4	0.2	0
17:48:06	24.2	56.4	92.4	81.2	79.8	79.5	78.2	77.9	77.3	76.6	76.0	75.9	75.9	75.9	27.5	29.7	13.9	14.4	0.4	0
17:48:36	24.2	55.7	92.3	80.5	78.8	78.4	77.4	77.0	76.7	75.7	75.1	74.9	74.9	74.9	27.5	29.6	13.9	14.4	0.2	0
17:49:06	24.3	55.1	92.4	80.2	78.2	77.8	76.5	76.2	75.1	74.3	73.2	74.0	74.0	74.0	25.6	29.4	14.1	14.5	0.5	0
17:49:36	24.2	54.3	92.1	79.8	77.2	77.0	75.7	75.6	74.8	73.6	72.9	73.3	73.3	73.3	26.4	29.3	13.9	14.6	0.2	0
17:50:06	24.1	53.8	92.2	79.7	76.6	76.3	75.0	74.7	74.2	72.9	72.4	72.6	72.6	72.6	29.2	14.1	14.1	14.6	0.6	0
17:50:36	24.1	53.2	92.1	78.9	75.9	75.4	74.3	74.0	73.6	72.3	71.9	71.9	71.9	71.9	26.7	29.1	14.1	14.7	0.6	0
17:51:06	24.1	52.3	92.1	79.4	75.4	74.7	73.5	73.5	72.7	71.6	71.0	71.0	71.0	71.0	27.0	29.1	14.3	14.9	0.6	0
17:51:36	24.2	51.9	92.0	79.5	74.6	73.8	72.7	72.7	72.7	72.1	71.0	70.4	70.4	70.4	27.1	28.9	14.4	14.9	0.0	0
17:52:06	24.2	51.3	92.1	79.4	74.2	73.0	71.9	72.1	71.3	70.2	69.8	69.6	69.6	69.6	27.2	28.8	14.5	15.1	0.2	0
17:52:36	24.1	50.7	92.0	79.1	73.7	72.5	71.3	71.3	70.6	69.7	69.2	68.8	68.8	68.8	27.2	28.8	14.6	15.2	0.5	0
17:53:06	24.2	50.2	91.8	78.9	73.3	71.7	70.6	70.8	69.9	68.9	68.6	68.2	68.2	68.2	27.3	28.9	14.7	15.4	0.5	0
17:53:36	24.2	49.7	91.9	78.7	72.8	71.1	69.9	69.9	68.9	67.8	67.3	67.5	67.5	67.5	28.0	28.8	14.9	15.5	0.5	0
17:54:06	24.3	49.1	92.0	78.4	72.5	70.5	69.3	69.2	68.2	67.1	66.7	66.9	66.9	66.9	18.2	15.0	15.0	15.6	0.5	0
17:54:36	24.2	47.5	91.8	78.4	71.6	69.9	68.8	68.6	67.8	66.7	66.1	66.1	66.1	66.1	26.2	17.4	15.2	15.8	0.7	0
17:55:06	24.1	45.6	91.9	78.5	70.8	69.1	68.1	68.0	65.9	65.8	65.6	65.6	65.6	65.6	16.3	15.3	15.9	15.5	0.2	0
17:55:36	23.9	43.6	91.8	76.9	70.2	68.6	67.3	67.4	66.6	65.6	65.3	64.9	64.9	64.9	15.6	15.5	16.0	16.0	0.4	0
17:56:06	23.7	41.7	91.6	76.8	69.7	67.7	66.9	66.7	65.9	65.0	64.4	64.4	64.4	64.4	21.8	15.5	16.0	16.5	0.5	0
17:56:36	23.3	40.1	91.8	77.1	69.2	67.0	66.0	65.9	64.3	63.9	63.8	63.7	63.7	63.7	27.0	22.3	15.6	16.2	0.2	0
17:57:06	23.0	38.7	91.8	76.9	68.9	66.7	65.6	65.4	64.7	63.7	63.4	63.1	63.1	63.1	26.8	22.9	15.9	16.3	0.2	0
17:57:36	22.8	37.4	91.8	76.9	68.6	65.8	64.8	64.8	64.2	63.2	62.9	62.5	62.5	62.5	26.9	22.6	16.0	16.3	0.4	0
17:58:06	22.4	36.3	91.8	76.5	68.5	65.3	64.4	64.2	63.5	62.6	62.3	62.0	62.0	62.0	27.0	20.7	16.1	16.5	0.4	0
17:58:36	22.3	35.2	91.8	76.6	68.0	64.7	63.7	63.6	62.9	62.0	62.0	61.3	61.3	61.3	27.0	20.5	16.1	16.6	0.2	0
17:59:06	22.0	34.4	91.6	76.1	67.2	64.1	63.1	62.5	61.4	61.5	60.9	26.9	26.9	26.9	20.5	16.2	16.7	0.2	0	100
17:59:36	21.6	33.3	91.6	76.6	66.7	63.6	62.6	62.5	61.8	60.9	61.0	60.4	60.4	60.4	20.2	16.3	16.8	0.2	0	100
18:33:36	25.7	25.9	72.7	57.5	46.7	40.3	39.0	39.1	38.9	38.7	39.3	25.3	25.3	25.3	22.3	20.9	21.6	0.5	0	100

Temperatures and differential pressure according to flow diagram / system diagram.
 Y1, Y2, Y3: Controller Outputs for heater, Reflux ratio, frequency of pump drive.

6.4.4.2 Self-recorded measured values (without PC assistance)

The self-recorded measured values are primarily the masses, densities and temperatures of the various mixtures/products. In the course of the experiment they were entered by hand on the "Continuous worksheet" (see Tab. 7.2, page 118).

These measured values are presented, together with the evaluation, in chapter 6.4.5.2, Tab. 6.18, page 105.

6.4.5 Evaluation

6.4.5.1 Presentation of automatically recorded measurement data (experiment aim 2)

The bases of the following diagrams are the measured value tables Tab. 6.15, page 99 to Tab. 6.17 . Please note that the diagrams have differing temperature and pressure ranges.

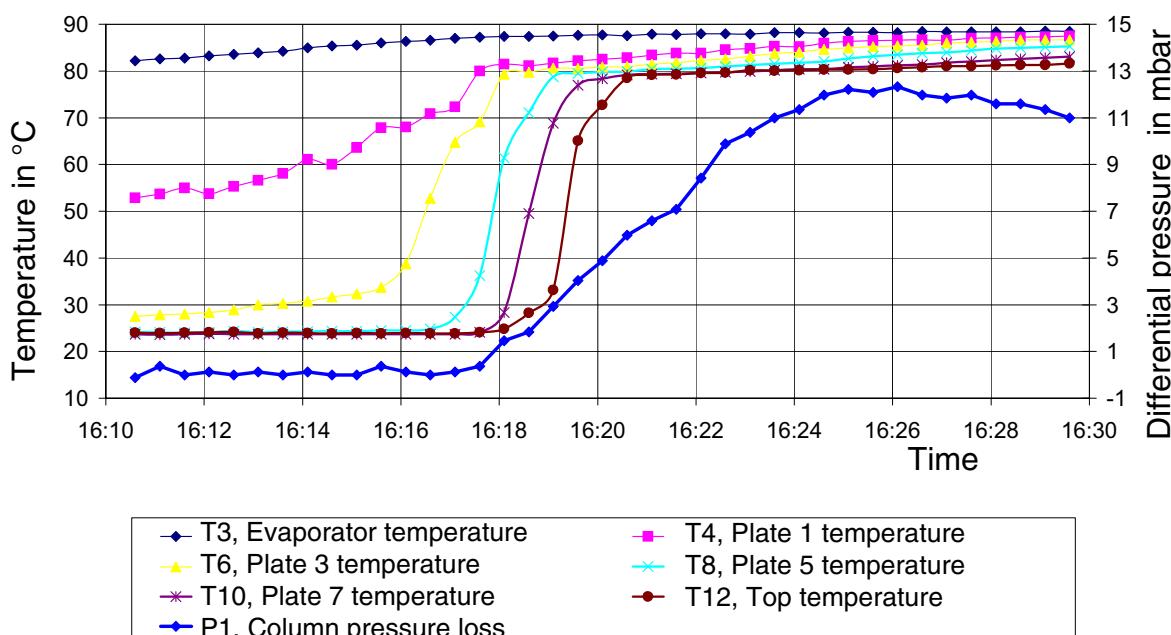


Fig. 6.6 V7, Temperature and pressure profile at column start-up

CE 600

CONTINUOUS RECTIFICATION

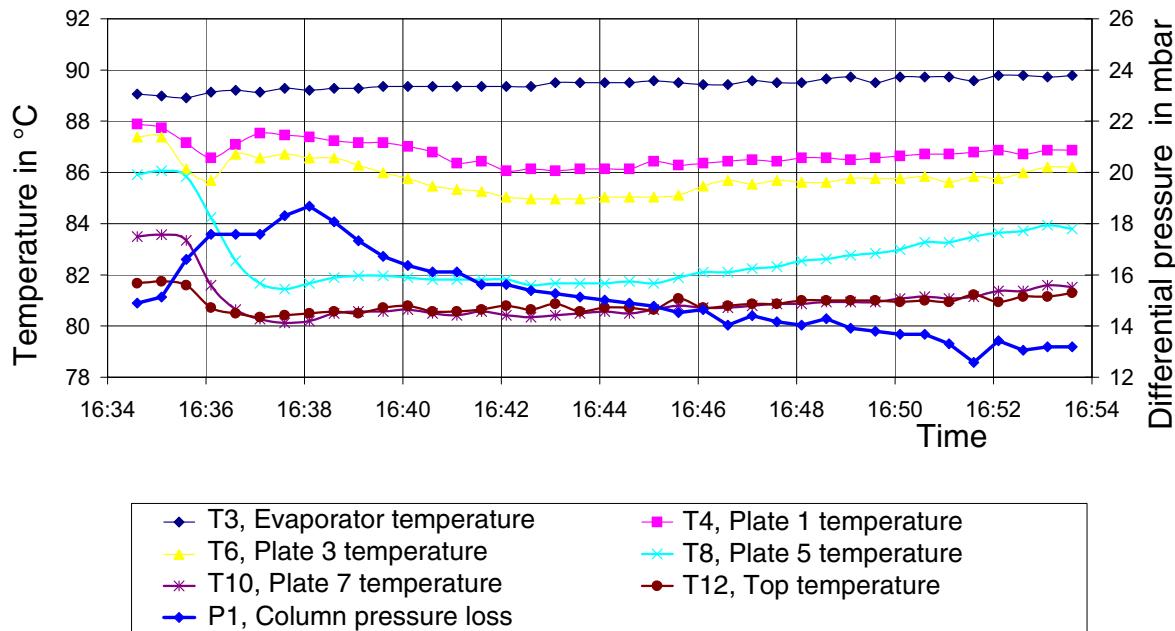


Fig. 6.7 V7, Temperature and pressure profile at start of rectification

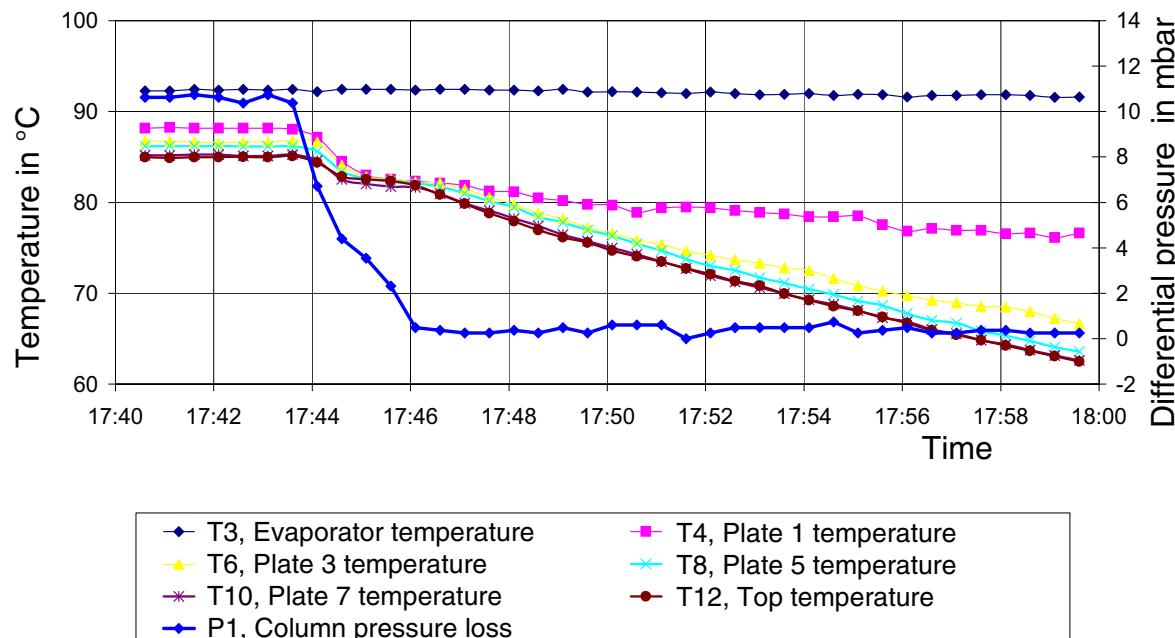


Fig. 6.8 V7, Temperature and pressure profile at end of rectification

6.4.5.2 Evaluation of self-recorded measured values

The following Tab. 6.18, page 105 presents the measured masses, densities and temperatures, together with the total masses and concentrations calculated from them.

The individual mixture masses at the start and end of the experiment have been added together (details follow in this chapter under item 5.).

Re experiment aim 3 - To determine the ethanol concentrations:

For all the samples, the ethanol mass fraction E_{w_E} was determined from the density and temperature by means of linear **interpolation** (for description of procedure see chapter 6.3.5.2, page 80).

The material values originate from Tab. 6.1, page 55 and Tab. 6.2, page 56.

Clock	Product/measurement no.	Measurement					Evaluation	
		Mass net g	Tempera-ture °C	Density kg/L	Level in Evap L	Comments	Mass net g	Mass fraction Ethanol, w _E %m
----	Start, mixture, Evap	----	24,0	0,9635	9,0		8744,5	22,3
16:52	B.pr., 1.	477,5	23,1	0,970	9,1		----	18,0
16:58	T.pr., 1.	402,5	23,5	0,825	9,1		----	86,1
17:02	B.pr., 2.	448,5	23,9	0,970	9,2		----	17,7
17:10	B.pr., 3.	430,0	24,3	0,970	9,1		----	17,6
17:16	T.pr., 2.	424,5	23,7	0,830	9,1		----	84,1
17:19	B.pr., 4.	475,0	25,0	0,970	9,1		----	17,4
17:29	B.pr., 5.	455,5	25,3	0,975	9,2		----	13,7
17:44	B.pr., 6. (+7.)	923,5	26,6	0,975	9,2	Total drainage	----	13,4
17:44	T.pr., 3.	636,5	23,5	0,835	----	Total drainage	----	82,2
----	End, T.pr., mixture	1620,0	23,0	0,833	----		----	83,1
----	End, B.pr., mixture	3204,5	24,2	0,972	----		----	16,2
----	End, B.pr., from Evap	----	21,8	0,977	----		8580,0	13,1

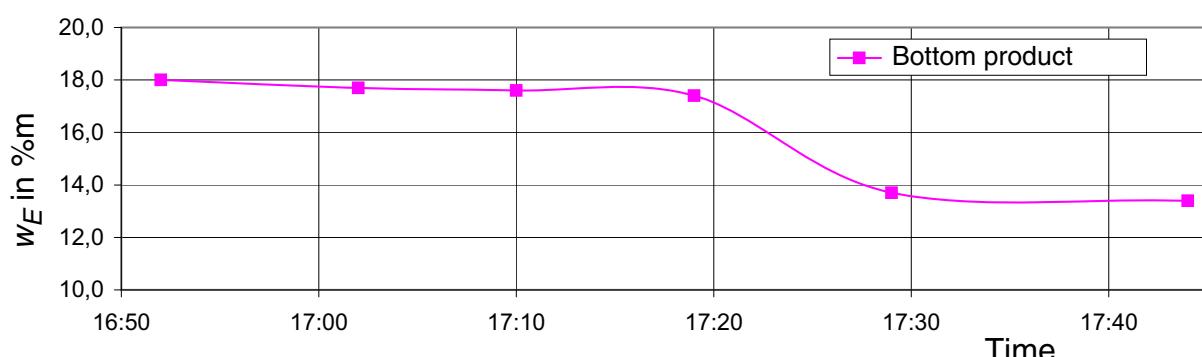
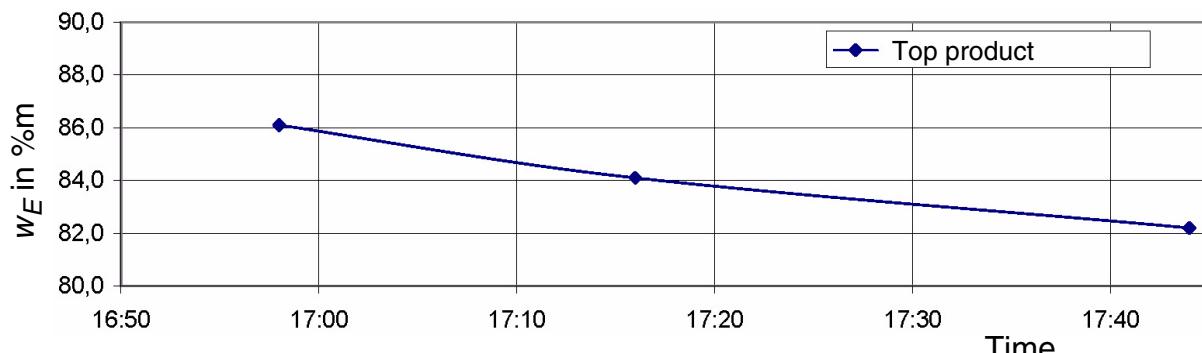
Measurement					
Start, Fill feed tank, Individual masses in g	1939,5	1939,5	1935,0	1929,5	1945,5 (-24,5g residue 1, when 10L reached)
Start, Fill feed tank, Individual masses in g	1941,0	1937,0	1944,5	1925,0	979,5 (24,5g see above; -7g residue 2, when 10L reached)
End, B.pr., from Evap, Individual masses in g	1930,0	1866,5	1855,0	1660,0	1268,5

Key: T.pr.: Top product; B.pr.: Bottom product; Evap: Evaporator

Tab. 6.18 V7, Self-recorded measured values, with evaluation

Re experiment aim 4. To present the change over time in the ethanol concentration in the top and bottom product

Fig. 6.9 shows the progression over time of the ethanol concentration:



Clock	Ethanol mass fraction	
	Top product w_E in %m	Bottom product w_E in %m
16:52	-----	18,0
16:58	86,1	-----
17:02	-----	17,7
17:10	-----	17,6
17:16	84,1	-----
17:19	-----	17,4
17:29	-----	13,7
17:44	82,2	13,4

Fig. 6.9 V7, Progression over time on the ethanol concentration in the top and bottom products

Re experiment aim 5. To check the concentrations by means of mass balances

It is useful to check the measured values with the aid of mass balances. Any conflicts arising from the mass balances indicate errors in evaluation or inaccurate measurement data.

At this point the mixture and ethanol masses at the start and end of the experiment are compared:

The following examples illustrate the graphical symbols used below:

$m:$	Mixture mass, general
$m_E:$	Ethanol mass, general
$w_E:$	Ethanol mass fraction, general
$m(Start):$	Mixture mass, at start of experiment
$m_E(End):$	Ethanol mass, at end of experiment
$m(Evap):$	Mixture mass in evaporator
$m_E(Feed):$	Ethanol mass in feed tanks
$w_E(T.pr.):$	Ethanol mass fraction in top product
$m(B.pr.):$	Mixture mass, bottom product

Mixtures mass balance:

To be checked:

$$m(Start) = m(End) \quad (6.6)$$

where

$$m(Start) = m(Start, Evap) + m(Start, Feed) \quad (6.7)$$

and

$$m(End) = m(T.pr.) + m(B.pr.) + m(B.pr., Evap) + m(End, Feed) \quad (6.8)$$

The following table illustrates how the values for $m(Start,Feed)$, $m(Start,Evap)$ and $m(B.pr.,Evap)$ are produced by adding together the individual mixture masses from Tab. 6.18, page 105:

	Mass, net, in g		
	$m(Start,Feed)$	$m(Start,Evap)$	$m(B.pr.,Evap)$
1. Measuring	1939,5	1941,0	1930,0
2. Measuring	1939,5	1937,0	1866,5
3. Measuring	1935,0	1944,5	1855,0
4. Measuring	1929,5	1925,0	1660,0
5. Measuring	1945,5	979,5	1268,5
Residue 1	-24,5	24,5	-----
Residue 2	-----	-7,0	-----
Total	9664,5	8744,5	8580,0

Tab. 6.19 V7, Addition of mixture masses

The “residue 1”, 24,5g, is included. This “residue 1” results from the fact that when transferring from the last measuring cup into the feed tank (VI), the total volume of 10,0L is reached before the measuring cup is completely empty. The residue is thus the mass of the mixture remaining in the measuring cup. After pumping the 9,0L into the evaporator, this “residue 1” is transferred into the feed tanks.

In the course of the experiment, 5,0L of the 10,0L feed is pumped into the column. The other 5,0L remain in the second feed tank. Consequently:

$$m(End,Feed) = m(Start,Feed) / 2 = \\ 9664,5 / 2 = 4832,3 \text{ g}$$

The following table consolidates the mixture masses calculated above and adds them together according to the formulae (6.7) and (6.8):

	Mass, net, in g	
	$m(Start)$	$m(End)$
$m(Start,Evap)$	8744,5	-----
$m(Start,Feed)$	9664,5	-----
$m(T.pr.)$	-----	1620,0
$m(B.pr.)$	-----	3204,5
$m(B.pr.,Evap)$	-----	8580,0
$m(End,Feed)$	-----	4832,5
Total	18409,0	18237,0

Tab. 6.20 V7, Mixture balancing

The comparison of the two values shows that at the end of the experiment there is still **99,1%** of the original mixture masses present.

Ethanol mass balance:

In line with the mass balances for the mixtures, (6.6) to (6.8), the resultant ethanol mass balances are:

To be checked:

$$m_E(Start) = m_E(End) \quad (6.9)$$

where

$$m_E(Start) = m_E(Start, Evap) + m_E(Start, Feed) \quad (6.10)$$

and

$$m_E(End) = m_E(T.pr.) + m_E(B.pr.) + m_E(B.pr.,Evap) + m_E(End, Feed) \quad (6.11)$$

The ethanol mass fraction w_E is defined as the ratio of the ethanol mass m_E to the mixture mass m . This enables the individual ethanol masses to be expressed as the product of the mixture mass and the mass fraction, e.g. for:

$$m_E(T.pr.) = m(T.pr.) \times w_E(T.pr.) \quad (6.12)$$

The following table summarises how the values from Tab. 6.18, page 105 and Tab. 6.20, page 109 are evaluated using the equations (6.10) to (6.12):

Designation ...	Start			End		
	$m(\dots)$	$w_E(\dots)$	$m_E(\dots)$	$m(\dots)$	$w_E(\dots)$	$m_E(\dots)$
g	%m	g	g	%m	g	
Start,Evap	8744,5	22,3	1950,0	-----	-----	-----
Start,Feed	9664,5	22,3	2155,0	-----	-----	-----
T.pr.	-----	-----	-----	1620,0	83,1	1346,0
B.pr.	-----	-----	-----	3204,5	16,2	519,0
B.pr.,Evap	-----	-----	-----	8580,0	13,1	1124,0
End,Feed	-----	-----	-----	4832,5	22,3	1077,5
Total	18409,0	-----	4105,0	18237,0	-----	4066,5

Tab. 6.21 V7, Ethanol balancing

The comparison of the two values shows that at the end of the experiment there is still **99,1%** of the original ethanol mass present.

6.4.6 Comments, assessment

The comments and assessment are based on the pre-defined experiment aims:

- Re 1. To concentrate the ethanol and
Re 3. To determine the ethanol concentration
in the initial mixture, top product and bottom
products**

The initial mixture was separated into the top product, with increased ethanol content, and the two bottom products, with reduced ethanol content.

The ethanol mass fraction in the top product is greater than in the initial mixture by a factor of **3,73**.

Comparison with the factor 3,51 from the batch experiment V3 (see also chapter 6.3.6, page 87) shows that in experiment V7 in continuous mode a better concentration was achieved.

- Re 2. To record the measured value using the data acquisition program (temperature and pressure profiles)**

Fig. 6.6, page 102 shows the profiles at column start-up. The curves run similarly to those in batch experiment V3.

The temperature rise in the individual plates from bottom to top is clearly traceable. Some of the temperature curves have a kink in them during heat-up. A kink of this kind can be interpreted as forming the equilibrium between vapour and liquid, on occurrence of the first condensate.

While the equilibriums are being created, the pressure loss in the column increases steadily.

The time between reaching the boiling temperature in the column head and the initial distillate in the phase separation tank (IV) is about 5 minutes under the selected conditions. When the first distillate occurs, the equilibria have formed and the differential pressure in the column now changes only to a minor extent.

Fig. 6.7, page 103 shows the differential pressure and temperatures at the start of rectification. Around 16:35 hours the operating conditions are adjusted (start pump at 20%, bottom feed, feed heating, start bottom product from evaporator to bottom product tank, reflux ratio R from 100 to 25%).

The many simultaneous changes take effect at different rates, and influence each other.

As a reaction, it is seen that the temperatures in the column initially fall - most rapidly in the upper section. At the same time the differential pressure rises. After about 10 minutes the complete system has adjusted to the new equilibria, and the temperatures slowly rise. The rise is in line with the rising boiling point at reduced ethanol concentrations.

Fig. 6.8, page 103 shows the profiles at the end of rectification. Around 17:44 hours operations are aborted by: Stop pump, stop bottom product from evaporator to bottom product tank, switch off heater.

The reaction of the temperatures in the column and column head is rapid. An initial rapid drop in temperatures by 2 to 5 °C is detected. At the same time the differential pressure falls, so the column loses condensate.

Compared to the corresponding profile in the batch experiment (see Fig. 6.3, page 79), the cooling process is slower here. However, the differential pressure in the column is reduced roughly twice as fast.

Re 4. To present the change over time in the ethanol concentration in the top and bottom product

Fig. 6.9, page 106 shows the decrease in the ethanol mass fraction w_E in the top product. It is seen that w_E decreases by 3,9 percentage points in the course of the experiment. This decrease corresponds to the result from batch experiment V3 (see chapter 6.3.6, page 87).

At first glance that appears a substantial decrease.

The reason for this decrease is that there is a greater proportion of ethanol in the newly created top product than in the bottom product of the evaporator. As a result, the bottom product in the evaporator loses ethanol in the course of the experiment. The decreasing concentration in the evaporator then results in a reduced concentration in the top product.

Fig. 6.9 also shows the decrease in the ethanol mass fraction w_E in the bottom product. As expected, w_E steadily decreases in the course of the experiment.

In the second half of the experiment - in the time band from 17:19 to 17:29 hours - a major decrease in w_E is seen. This particular curve trend should not be expected by way of reproducibility in any subsequent repetitions of the experiment. Alongside the known inaccuracies of the density measurement, there is another reason for variations here:

The measured bottom product flows permanently out of the evaporator. This discharging bottom product is always a mixture of existing evaporator content and newly arriving, differently concentrated condensate from the column. The intermingling of the evaporator content with the condensate varies in intensity.

Re 5. To check the concentrations by means of mass balances

Both regard to the mixtures and to ethanol, comparison of the two values shows that at the end of the experiment there is still **99,1%** of the original mass present (see above).

In view of the losses during sampling and transfer, and due to evaporation, these values are of a realistic magnitude.

This confirms the measured values and the evaluation of the experiment.

7 Appendix

7.1 Technical data

Overall unit:	<i>l x w x h :</i>	1300 x 750 x 2300 mm
Weight:		approx. 210 kg
Electrical supply:		3 x 400V / 50 Hz Optional alternatives, see rating plate
Cooling water flow:		min. 400 L/h
In-house water supply (p ≥ 1,5 bar g, T ≤ 25°C)		
Sieve plate column:	<i>Ø x h</i>	50 x 765 mm
	Material:	Stainless steel
	Number of plates:	8
Packed column:	<i>Ø x h</i>	50 x 765 mm
	Material	Stainless steel
	Packing	Raschig rings
	Material	Soda glass
	<i>Ø x h</i>	4 x 4 mm
Evaporator:	Heating power	4,0 kW
	Capacity	max. 10 L
	Minimum fill	3 L
Feed pump:	Capacity	approx. 0 ... 400 ml/min
	Internal electrical supply	24 VDC
Temperature sensors T1 ... T16 : Pt100		0...150 °C
Differential pressure sensor: piezoresistive		0 ... 0,25 bar
Experiment mixture:	ethanol/water	~ 25 : 75 %m

Volumes:	Feed tanks	~ 5 L
	Bottom product tank	~ 5 L
	Top product tank	~ 1,5 L
	Phase separation tank	~ 0,5 L
	Solvent tank	~ 0,9 L
Data acquisition:	USB communication	
	Program environment: LAB-VIEW Runtime	
	System requirements:	
	• PC with Pentium IV processor, 1 GHz	
	• Min. 256 MB RAM	
	• Min. 250 MB available memory on hard disk	
	• 1 x USB port	
	• Graphics card resolution min. 1024 x 768 pixels, True Color	
	• Windows 2000 / XP / Vista	

7.2 Worksheets

The following pages set out a series of worksheets. The worksheets can be used to enter self-recorded measurements by hand during experiments.

The “Batch” worksheet in Tab. 7.1 can be used for experiments from the “Batch” series.

The “Continuous” worksheet in Tab. 7.2 is intended for experiments in the “Continuous” series.

Experiment no.:		Date:		Participants:					
Clock	Product/measurement no.	Measurement					Comments	Evaluation	
		Mass net g	Tempera-ture °C	Density kg/L	Level in Evap L	Mass net g		Mass fraction Ethanol, w_E %m	
	Start, mixture, Evap	-----							
	T.pr., 1.							-----	
	T.pr., 2.							-----	
	T.pr., 3.							-----	
-----	End, T.pr., mixture				-----			-----	
-----	End, B.pr., Evap	-----			-----				

Measurement					
Start, Fill feed tank, Individual masses in g					
End, B.pr., from Evap, Individual masses in g					

Key: T.pr.: Top product; B.pr.: Bottom product; Evap: Evaporator

Tab. 7.1 "Batch" worksheet for self-recorded measurements in the "Batch" experiment series

Experiment no.:		Date:		Participants:				
Clock	Product/measurement no.	Measurement				Comments	Evaluation	
		Mass net g	Tempera-ture °C	Density kg/L	Level in Evap L		Mass net g	Mass fraction Ethanol, w_E %m
	Start, mixture, Evap	-----						

-----	End, T.pr., mixture				-----		---	
-----	End, B.pr., mixture				-----		---	
-----	End, B.pr., from Evap	-----			-----			
Measurement								
Start, Fill feed tank, Individual masses in g								
Start, Fill feed tank, Individual masses in g								
End, B.pr., from Evap, Individual masses in g								

Key: T.pr.: Top product; B.pr.: Bottom product; Evap: Evaporator

Tab. 7.2 "Continuous" worksheet for self-recorded measurements in the "Continuous" experiment series

7.3 Abbreviations and symbols

7.3.1 Abbreviations used

B.pr.	Bottom product
B.pr.tank	Bottom product tank
Evap	Evaporator
HTU	Height of Transfer Unit
LSL	Level Switch Low
NTU	Number of Transfer Units
Pd	Packed column
SP	Sieve plate column
T.pr.	Top product
T.pr.tank:	Top product tank

7.3.2 Symbols used

f	Frequency	Hz
m	Mass, mixture mass	g, kg
m_E	Ethanol mass	g, kg
p	pressure	bar, mbar
P_{el}	Electrical Power Output	kW
R	Reflux ratio	%
t	Time	min, s
T	Temperature	°C
V	Volume	L
\dot{V}	Volumetric flow	L/h
w	Mass fraction	%m
w_E	Ethanol mass fraction	%m
x_1	Molar fraction in the liquid mixture	%
y_1	Molar fraction in the steam	%
ρ	Density	kg/L, kg/m³

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