BE16 Practical Training CRE 1 Residence Time Distribution – Real Reactor Behaviour			HOCHSCHULE RHEIN-WAAL Rhine-Waal University of Applied Sciences
Laboratory	Life Sciences	Process Engineering	Version 1.0_2023
Instructors	Prof. Dr.-Ing. Frank Platte	Dipl.-Ing. Carlos Perez-Bolde	

Last Name, First Name (*Please write legibly*)	**Student ID**	**Student's Signature**	**Entry test passed**	**Instructor's Signature**
1.				
2.				
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4.				

Introduction and theory

The residence time - also called age - indicates how long a particle, a molecule or a fluid package remains in a test volume. In general, these test volumes could have numerous in- and outflows. Only in an ideal setup all molecules or fluid parcels inside a test volume could have the same age, but in general there is a so-called Residence Time Distribution (RTD).

The concept of residence time originated in models of chemical reactors, but it can also be applied to geochemical reservoirs or drugs in a human body, smog atmosphere in cities e.g. Los Angeles and many more. If you conduct a survey in a process engineering lecture, asking from which semester the students are attending the lecture, you will probably get a result similar to the one shown below. Some brave students might already join the lecture in the first semester, whereas others are from higher semesters 5, 7 and 9. The majority of the students follow the official curriculum and participate in the 3rd semester. By dividing all numbers by 74 (total number of students) you can easily get the percentage of each class.

Semester	Number of answers	%
1	2	2.7
3	50	67.6
5	15	20.3
7	6	8.1
9	1	1.4

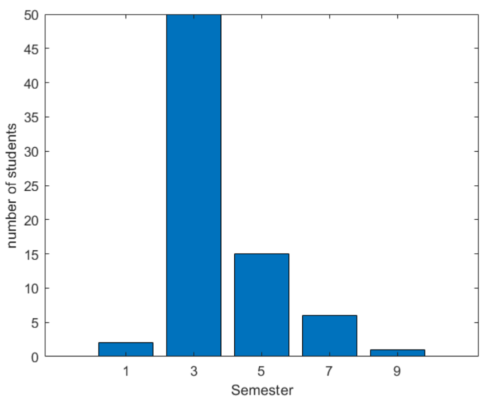


Figure 1: Residence time Distribution (RTD) of students in a lecture.

In order to measure the Residence Time Distribution, E(t), of a chemical reactor, you need to use a tracer. A tracer is a substance that is added to the primary fluid and that ideally has the same flow behaviour as the primary fluid in the reactor. Very often either saline solution (changing the conductivity) or dyes (changing the colour of the fluid) are used as tracers. In this practical training we use a NaCl solution. The concentration of salt is measured by means of a conductivity sensor.

The following figure illustrates the two most commonly used *input signals*, i.e. the step and the Dirac pulse, in order to derive the RTD. Theoretically, the Dirac pulse is the ideal input signal since its response is (after normalizing) exactly the E(t) curve. Alternatively, a step can be applied as input signal resulting, also after normalizing, in a F-type curve, F(t). Any other input signal is modulated (convoluted) through the weighting function E(t). To get E(t) from arbitrary input signals sophisticated deconvolution procedures must be deployed on the measured output values.

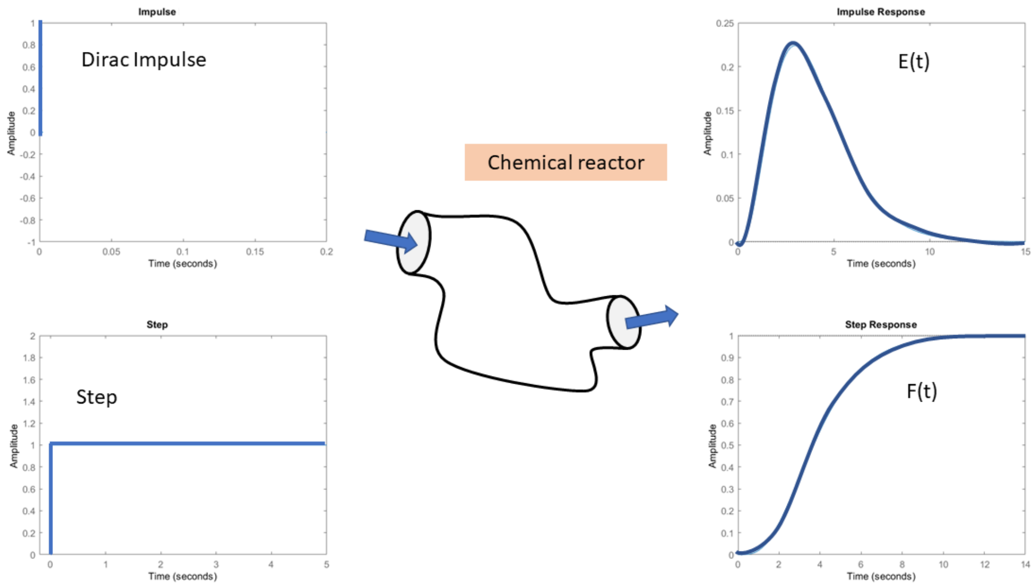


Figure 2: Connection between E(t) and F(t) curve as well as for Dirac pulse and a step function.

As stated above, the E(t)-curve can be generated with a Dirac pulse if you wait an infinitely long time, so that all the particles fed into the inlet be washed out of the system. As a consequence, the following definition makes sense:

$$\int_0^\infty E(t)dt = 1. \qquad (1)$$

In other words, formula (1) means that the probability for a particle to leave the reactor between the beginning of the experiment and any time in the future is 100% (i.e. 1). Beside the E(t)-curve, another curve is frequently used: The F(t)-curve. The F(t)-curve sums up all classes up to the age *t*. Mathematically the connection between these two curves is as follows:

$$F(t) = \int_0^t E(t)dt \qquad (2)$$

with F(0) = 0 and F(t $\rightarrow\infty$) = 1. Given a F(t)-curve i, the RTD can be simply derived by differentiation:

$$\frac{dF(t)}{dt} = E(t) \qquad (3)$$

A step is *experimentally* much easier to set up than a Dirac pulse. That is why we record the F(t) function in this practical training, and calculate E(t) with formula (3). There are two statistical quantities that are usually extracted from the measured data. These are the first and second moments, also referred as to the Mean residence time, $\bar{t}$, and the Variance, σ^2, that is the square of the standard deviation, σ.

$$\bar{t} = \frac{\int_0^{\infty} t\, E(t) dt}{\int_0^{\infty} E(t) dt} \approx \frac{\sum_i t_i\, E(t_i)}{\sum_i E(t_i)} \qquad (4)$$

$$\sigma^2 = \frac{\int_0^{\infty} (t-\bar{t})^2\, E(t) dt}{\int_0^{\infty} E(t) dt} \approx \frac{\sum_i (t_i-\bar{t})^2\, E(t_i)}{\sum_i E(t_i)} \qquad (5)$$

Ideal reactor behaviour

The simplest case is the Batch reactor (BR). The operator decides when the experiment starts and stops. Since a BR is a closed system, all the molecules have the same age, but might change their identity due to a reaction. More interesting are the continuously operated reactors, such as the Plug-flow reactor (PFR) and the Continuously stirred tank reactor (CSTR) reactor. The following figure illustrates the expected ideal behaviour of a CSTR and a PFR. Due to its perfect back-mixing, the CSTR washes out the traces continuously leading to an exponential decay. In the case of an ideal PFR, the response is shifted by the Space time, τ. In control engineering these responses of the system are respectively called First order lag element and Dead time behaviour.

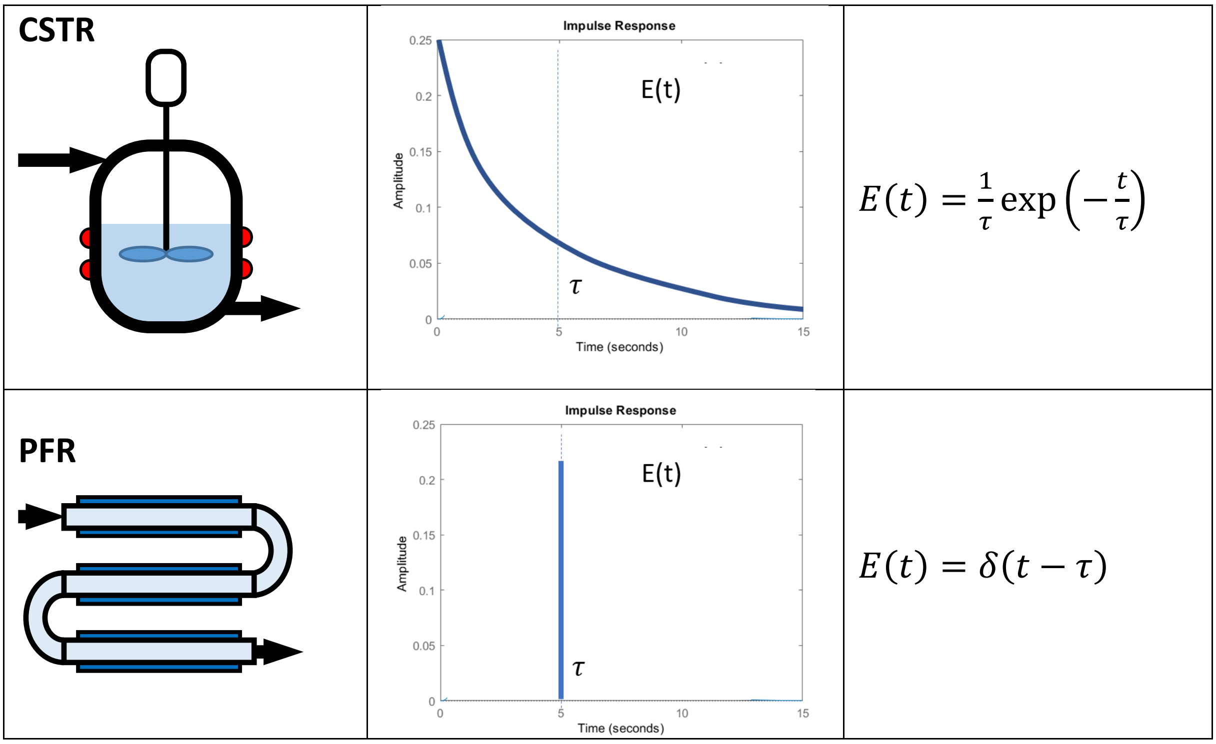
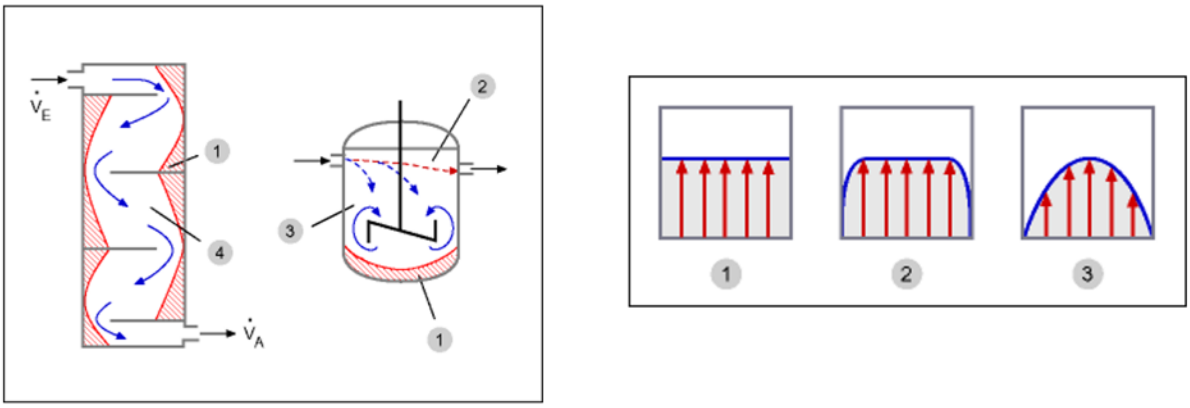


Figure 3: Theoretical behaviour of the ideal CSTR and the ideal PFR.

Reasons for non-ideal flow

Real reactors might exhibit Stagnant zones (dead zones), Short circuits and Channelling. As a consequence, their behaviour might significantly deviate from ideal reactors. Additionally, a plug flow is nearly impossible to establish. This is because the flow profile within tubular reactors, both in laminar or turbulent flows, shows a velocity distribution along the cross section. Thus, the molecules reach the outlet of the reactor at different times.

What makes a reactor non-ideal?



"Dead Zone" (1),
"Short Circuit" (2)
ideal mixing (3)
ideal plug flow (4)

ideal plug flow (1).
(2) turbulent flow
(3) laminar flow

Figure 4: Some effects that lead to non-ideal behaviour in real reactors. (Source: Chemgapedia)

To date measuring the RTD of real reactors is a powerful tool for characterising the behaviour of a system. Analysing the RTD helps detecting “illnesses” (e.g. channelling, short circuits, stagnant zones) of a chemical reactor. A rigorous analysis of the flow using computational fluid dynamics (CFD) may lead to more detailed results.

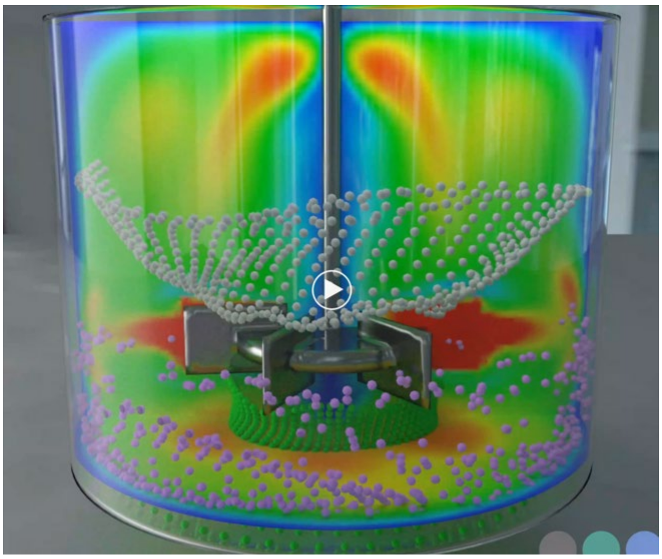


Figure 5: CFD Simulation of a CSTR. (Source: Dr. Mierka, IANUS Simulation GmbH)

Prerequisites for participating in the practical training

In order for you to be allowed to take part in the practical training you have to fulfil ALL of the following:

- Have successfully passed the (general) Safety briefing course on Moodle, and be in possess of a valid lab ID card.

- Wear a lab coat (please bring your own), closed shoes and clothing that <u>completely</u> covers the legs. We have nothing against mode, but in chemical labs and facilities **safety has priority**!

If you happen not to comply with the previous requisites, you will not be allowed into the lab. NO EXCEPTION WILL BE MADE.

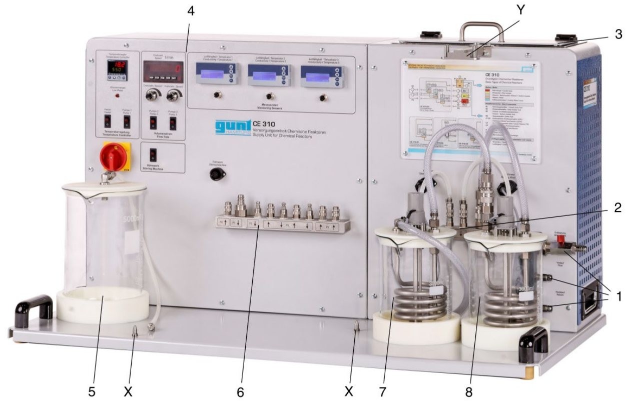
All the other safety equipment will be provided to you.

Additional information on the safety equipment in our lab is available on the short **<u>video "BE16 Lab 01 – Safety & Preparation."</u>** Please watch it.

- Furthermore, in order for you to be able to conduct the practical training, you must pass a pre-practical training exam. This is conducted verbally on-site and is based on:
    - The information in this document,
    - The Preparation Questions available at the end of this document and in Moodle, and
    - The short videos available on Moodle that we prepared for you to get the most out of the practical training.

Shall anything else be needed or if you happen to have any questions, please do not hesitate to contact us.

Unit layout and function



1 Connection strip for cold water and drain
2 Connection strip for educts and water
3 Water tank
4 Control panel and electrical module connection
5 Product tank
6 Module connection strip
7 Educt tank (caustic soda, educt A)
8 Educt tank (ethyl acetate, educt B)
X Positioning pins
Y Mount

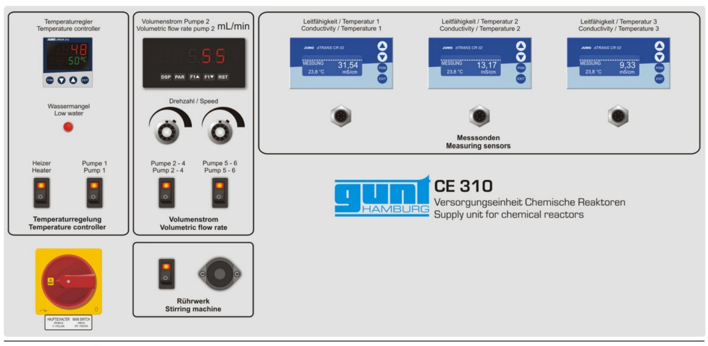
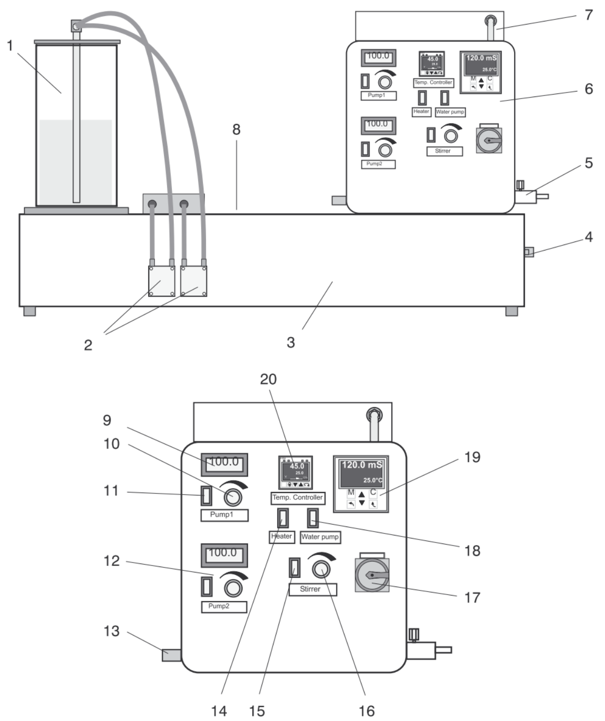


Figure 6: Chemical reactor CE310 new version.



1 Tank
2 Pump
3 Base of the device
4 Sleve terminals for CE 310.03
5 Water drain cock
6 Switch box
7 Water tank
8 Special steel trough
9 % Display Pump 1
10 Speed Pump 1
11 On/ Off switch Pump 1
12 Pump 2 analogous to Pump 1
13 Water connections
14 Switch for heater
15 Switch for stirrer
16 Speed stirrer
17 Master switch
18 Water pump
19 Measurind device for temperature and conductivity
20 Temperatur controller

Figure 7: Chemical reactor CE310 old version.

Further information on the Chemical Reactor Systems that you will be using is respectively available on the **videos "BE16 Lab 02 - The Chemical Reactor System"** and **"BE16 Lab 03 - Process Flow Diagram & Equipment Operation."** Please watch them.

Relevant technical data

The measurements of these practical training rely on the conductivity of aqueous substances. Table 1 shows the electrical conductivity of the three substances that will be used in this practical training, i.e. deionised water, tap water and a table salt (sodium chloride, NaCl) aqueous solution.

Table 1. Electrical conductivity of the substances to be used

Substance	Concentration [M]	Electrical conductivity (λ) [mS/cm]
Deionised water	-	~ 0
Tap water (our lab in Kleve)	-	0.29 - 0.45
Sodium chloride (NaCl) solution	0.2	> 16

The required solutions for the first experiment will be provided to you, so that you can focus on learning how to operate the reactors, and conduct straightway the first step-response experiment. However, you will have to prepare your own solutions for the second experiment.

As you do not know which reactor you will be using, **calculate** and **bring the answer to the pre-lab test**. What amount of educts do you need to prepare the following solutions?

a) 2,0 L NaCl 0,1 M
b) 0,5 L NaCl 0,2 M
c) 1,0 L NaCl 0,2 M

Assume that the sodium chloride (NaCl) is 100% pure salt; its molecular weight is 58.443 g/mol.

Further information on how to prepare the solutions, on the influence of volume in their conductivity and on the response time of the sensors that you will be using is available on the **video "BE16 Lab 04 - Solutions, Reactants & Conductivity."** Please watch it.

Activities

This practical training consists of the following activities, and is planned to be conducted in 95 minutes.

1. Safety briefing
2. Pre-Lab Test
3. Getting to know the reactors, initial setup and checkup
4. Experiment 1
5. Flushing of the reactor
6. Preparation of the solutions for Experiment 2
7. Experiment 2
8. Flushing of the reactor
9. Cleaning the laboratory (i.e. leaving it as you found it).

Shall everything proceed properly; the instructor will sign the cover page of this instruction's manual.

Do not forget to print the present instruction's manual, and bring it to the practical training!

Please also bring a USB-Stick, as you will need it to unload your data, and a stopwatch.

Tasks

The **video “BE16 Lab 05 - Residence Time Distribution”** shows how to conduct, among other things, the experiments described below so that you have a head start. Please watch it.

1. Switch on the GUNT CE 310 reactor that was assigned to your group.

Software check and setup

2. Open the GUNT software according to the version (old, new) of your assigned CE 310 reactor. You can find the respective shortcuts on the computer’s desktop. Double click on them.

(Old CSTR) 

(New CSTR, new PFR) 

3. Check that the diagram shown by the software corresponds to your assigned CE 310 reactor i.e. PFR or CSTR (Fig. 8). If not, please, contact a supervisor.

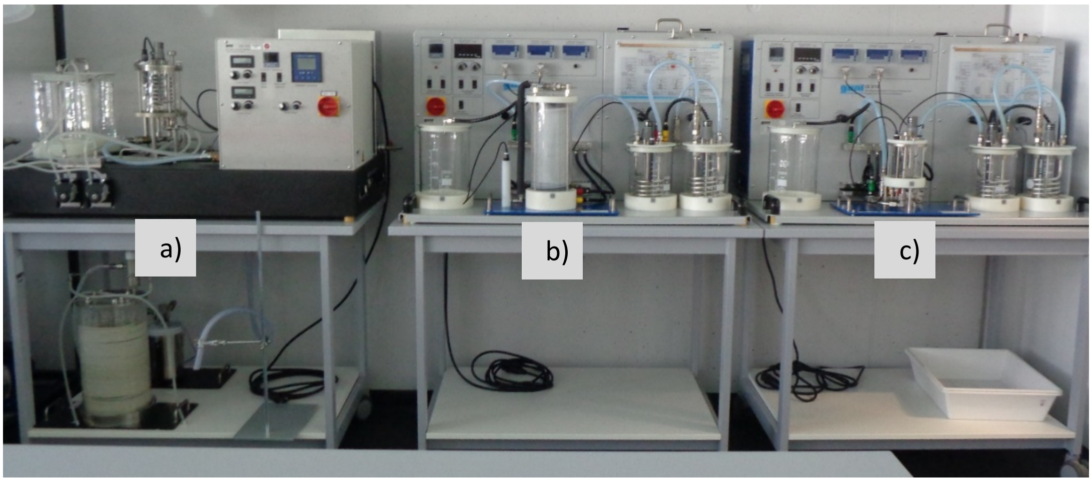


Figure 8: Chemical reactors: a) CSTR (old version), b) PFR (new version), and, c) CSTR (new version).

4. Complete the software settings of your assigned CE 310 reactor according to Table 2.

Table 2. Software settings		
CSTR (old version)	**PFR (new version)**	**CSTR (new version)**
• Start > Charts • Click on: Settings • Set “Interval” from 60 **to 1** s • Click on: Indicator • File > New • Give a name to your file, choose “Desktop” to save it • Click on: “OK” • Minimize the window (do not close the software)	• Close the software	• Close the software

First experiment setup

5. Depending on the CE 310 reactor assigned to you, set the parameters for the 1st experiment as shown in Table 3.

Important

Only set the parameters; **do not start anything yet!**

Table 3. Setup of the 1st experiment.

Device	**CSTR (old version)**	**PFR (new version)**	**CSTR (new version)**
Tank B1	1,5 L H_2O	0,5 L H_2O	0,5 L H_2O
Tank B2	2,0 L NaCl **0,1 M**	1,0 L H_2O	1,5 L H_2O
Pump 1	70%	---	---
Pump 2	70%	50%	50%
Stirring	Yes, 100%	Yes	Yes
Reactor volume	890 mL (already adjusted)	290 mL (the volume is fix)	290 mL (already adjusted, 4.4-cm-high)
Cooling water pump	No	Yes	No
Temperature	Room temperature	Room temperature	Room temperature

Safety check-up

6. Check all of the following:

Important

When you work with chemicals or with chemical equipment, you do it as a GROUP. This means that **you are not only responsible for your own safety**, but **also for your teammates' safety**!

This includes that everyone wears the safety equipment.

Safety equipment

Are you and your teammates wearing…

- Lab coat?
- Safety googles?
- Closed shoes?
- Long pants?
- Are people with long hair keeping it up?
  (This is important due to the rotating shafts of the stirring devices)
- Earplugs?
- Are the earplugs correctly been used? **(The sounds around you should be muffled!)**

Unfortunately, the CE 310 reactors are very loud. This is why **<u>it is a requirement to use earplugs during this practical training</u>**. Failing to do so or removing them while the CE 310 reactors are in operation, will result in your exclusion from the laboratory. In fact, due to the sound levels, we are required by law to control the use of hearing protection devices.

<u>Equipment</u>

- Is the reactant's sensor in tank B1?
- Is the reactor's sensor in the reactor?
- Is the piping correctly and properly attached?
- Are all the required valves open?
- Do the reactants' tanks have their cover on?
- Does the products' tank have its cover on?
- Is every surface of the reactor dry?

Important: Shall any liquid spill out of the reactor during its operation, the reactor must be shut down immediately**.**

<u>Substances</u>

- Do the solutions have the concentration specified in Table 3?
- Are the solutions in the respective tanks according to Table 3?
- Is the solution for the step-response already prepared?
- Is the solution for the step-response readily available?
- Is the solution for the step-response away from electrical cables?

<u>Experiment 1</u>

7. Start the stirring and the cooling water pump according to your assigned reactor *and* Table 3.

8. Start the reactant's pump: **a)** old CSTR: *Pump 1***; b)** new PFR or new CSTR: *Pump 2*.

9. Wait until the **<u>first drop of liquid gets into the product's tank</u>**, this is time **t=0**
    - Start your stopwatch and, simultaneously,
    - <u>Start recording your data</u> as indicated on Table 4.

Table 4. Procedure to start recording data on the different CE 310 Reactors

CSTR (old version)	**PFR (new version)**	**CSTR (new version)**
• Maximize the window  • Start > Charts • Click on:  • Start > Charts	• Start the GUNT software (Double-click on the shortcut on the desktop) • View > Time Curve	• Start the GUNT software (Double-click on the shortcut on the desktop) • View > Time Curve

10. **“Step-Procedure”**

- Conduct the “Step-procedure” according to indications in Table 5 depending on the CE 310 Reactor assigned to you.

<table>
<tr><th colspan="3">Table 5. Conduction of the “Step-procedure” in experiments 1 and 2.</th></tr>
<tr><th>CSTR (old version)</th><th>PFR (new version)</th><th>CSTR (new version)</th></tr>
<tr><td rowspan="3">• At time t_0 + 1.5 minutes
(After 1 Minute 30 seconds)
Simultaneously:
○ Turn off Pump 1
○ Turn on Pump 2</td><td colspan="2">• Wait until the electrical conductivity in reactant’s tank B1 starts to decrease (reading on ___▪___), and</td></tr>
<tr><td>▪ <u>Sensor 1 (new PFR)</u></td><td>▪ <u>Sensor 3 (new CSTR)</u></td></tr>
<tr><td>○ Pour into tank B1
0,5 L NaCl 0,2 M</td><td>○ Pour into tank B1
1,0 L NaCl 0,2 M</td></tr>
<tr><td colspan="3">• Write down the time (<u>t_{step}</u>) in the time log [Annex 1]</td></tr>
<tr><td><u>t_{step}</u></td><td colspan="2"><u>t_{step}</u> or, in this case, also <u>$t_{pouring}$</u>
• Make a note on the approximate time count on the software</td></tr>
</table>

11. **Time of first (sensor) reaction**

- When the electrical conductivity in the reactor starts to increase (reading on ___◦___), it is the time of first (sensor) reaction,
  - <u>Sensor 5</u> in the old CSTR
  - <u>Sensor 2</u> in the new PFR
  - <u>Sensor 4</u> in the new CSTR
- Write down the time of first (sensor) reaction ($t_{First\ Reaction}$) in the time log [Annex 1].
- (New CSTR, new PFR) Make a note on the approximate time count on the software.

12. **Printouts to be included in the report**

- When the reactants are almost depleted (visual control), make a screenshot of:

i. The System Diagram.	Old CSTR:	Start > System Diagram
	New PFR, new CSTR:	View > System Diagram
ii. The Time Curve.	Old CSTR:	Start > Charts
	New PFR, new CSTR:	View > Time Curve

- Save these printouts as images, print them, and include them in your report.
- Generate (“print”) a pdf-graph of the time curve from the Gunt Software on the computer’s Desktop:

Old CSTR: File > Print

New PFR, new CSTR:  > Adobe PDF >  > “OK”

13. **Experiment's end**

- When the reactants' tanks are depleted, the experiment has come to its end. Proceed as indicated in Table 6.

<table>
<tr><th colspan="3">Table 6. End of the experiment (experiments 1 and 2).</th></tr>
<tr><th>CSTR (old version)</th><th>PFR (new version)</th><th>CSTR (new version)</th></tr>
<tr><td colspan="3">• When the reactants' tanks are depleted (Indicators: ◊)</td></tr>
<tr><td>◊ Visual control</td><td colspan="2">◊ Visual control, and
◊ The electrical conductivity in reactant's tank B1 starts to decrease (reading on ▪)</td></tr>
<tr><td>○ Turn off Pump 2</td><td>▪ Sensor 1 (new PFR)
○ Turn off the stirring
○ Turn off the cooling water pump
○ Leave Pump 2 on (!)</td><td>▪ Sensor 3 (new CSTR)
○ Turn off the stirring
○ Turn off Pump 2</td></tr>
<tr><td colspan="3">• Write down the time (t_{end}) in the time log [Annex 1]</td></tr>
<tr><td>t_{end}</td><td colspan="2">t_{end}
• Make a note on the approximate time count on the software</td></tr>
</table>

Before shutting off the reactor save your data, and check that it was correctly saved!

14. **Data saving on the Gunt Software**

- To save or export data from the Software, follow the steps on Table 7 depending on the CE 310 Reactor that you used.

<table>
<tr><th colspan="3">Table 7. Saving or exporting data from the Gunt Software.</th></tr>
<tr><th>CSTR (old version)</th><th>PFR (new version)</th><th>CSTR (new version)</th></tr>
<tr><td>• Check that your data was correctly saved
[Your file is on the desktop with text (*.txt) format]</td><td colspan="2">• View > Time Curve
•  >  > Give a name > "OK"
• Double-click on the "Gunt-Data"-shortcut on the Desktop
• Check that the data was correctly saved</td></tr>
</table>

15. **Cleaning and flushing the reactor**

- Turn the CE 310 Reactor off, and write down the time in the time log [Annex 1].

  Hint: To be sure that no salt is being used while flushing the reactors, the first thing to be done is washing twice (2 times) the containers that you will use wash, and fill the reactants' tanks.

- Rinse twice (2 times) with water

  i. The sensors, and

  ii. The reactant's tank where the salt solution was.

- Follow the instructions of your supervisor to flush your assigned CE 310 reactor. The respective pump power and amounts of water to be used are in Table 8.

Table 8. Pump power and water required to flush the CE 310 reactors.			
Device	**CSTR (old version)**	**PFR (new version)**	**CSTR (new version)**
Reactant's Tank B1	1,5 L H_2O	0,7 L H_2O	1,2 L H_2O
Reactant's Tank B2	---	0,7 L H_2O	1,2 L H_2O
Pump 1 [%]	90	---	---
Pump 2 [%]	0	90	90

Experiment 2

Redo the experiment with a larger volumetric flow rate. To do this:

16. Prepare the required salt solution according to your CE 310 reactor and Table 9. You only need to prepare the solution for your reactor (not all 3 of them)

Table 9. Solutions required for conducting the second experiment.		
CSTR (old version)	**PFR (new version)**	**CSTR (new version)**
2,0 L NaCl **0,1 M**	**0,5 L** NaCl **0,2 M**	**1,0 L** NaCl **0,2 M**

17. Close the software, and start from the very beginning.
18. The setup of the second experiment can be found in Table 10.

Table 10. Setup of the 2nd experiment.			
Device	**CSTR (old version)**	**PFR (new version)**	**CSTR (new version)**
Tank B1	1,5 L H_2O	0,5 L H_2O	0,5 L H_2O
Tank B2	2,0 L NaCl 0,1 M	1,0 L H_2O	1,5 L H_2O
Pump 1	90%	---	---
Pump 2	90%	70%	70%
Stirring	Yes, 100%	Yes	Yes
Reactor volume	890 mL (already adjusted)	290 mL (the volume is fix)	290 mL (already adjusted, 4.4-cm-high)
Cooling water pump	No	Yes	No
Temperature	Room temperature	Room temperature	Room temperature

19. All the other steps are the same as in experiment one.
20. Do not forget filling in the time log, doing the screenshots, and saving the data.
21. Name respectively the two text (*.txt) files with your results “CRE1 - Team _#_ - Run _*_”, where # is your teams number and * the run i.e. either 1 or 2.
22. **<u>Leave the two files of the previous point on the desktop of the computer used</u> (Failing to do it, may result in failing the practical training!)**
23. Download all your files onto your USB-Stick.
24. Turn off your CE 310 reactor, and leave it clean and dry (i.e. leave it as you found it)
25. Ask the supervisor to check it, and to sign the cover page of this manual.

26. Prepare your report. The guidelines are in Annex 2.

Annex 1. Time Log

Group: _________ **Reactor used:** _________________ **Date, Time:** _________________

First Experiment

Pump(s) [%]:________ **$\lambda_{max,\ Tank\ B1}$ [mS/cm]:**________ **$\lambda_{max,\ Reactor}$ [mS/cm]:**________

Event		**Time** [MM:SS]	**Time count on the software (approx.)** (only for the new reactors)
Start time i.e. 1st drop in the products' tank	$t = 0$	0:00	
Step-Procedure	t_{step} or $t_{pouring}$		
Time of first (sensor) reaction	$t_{First\ Reaction}$		
Experiment's end	t_{end}		
Reactor shut off	t_{off}		

File check:

Time curve	(Screenshot):	Yes / No	System Diagram	(Screenshot):	Yes / No
Time curve	(pdf):	Yes / No	Data file	(txt):	Yes / No

Second Experiment

Amount of salt (NaCl) you used for preparing the second-experiment solution [g]: ______

Pump(s) [%]:________ **$\lambda_{max,\ Tank\ B1}$ [mS/cm]:**________ **$\lambda_{max,\ Reactor}$ [mS/cm]:**________

Event		**Time** [MM:SS]	**Time count on the software (approx.)** (only for the new reactors)
Start time i.e. 1st drop in the products' tank	$t = 0$	0:00	
Step-Procedure	t_{step} or $t_{pouring}$		
Time of first (sensor) reaction	$t_{First\ Reaction}$		
Experiment's end	t_{end}		
Reactor shut off	t_{off}		

File check:

Time curve	(Screenshot):	Yes / No	System Diagram	(Screenshot):	Yes / No
Time curve	(pdf):	Yes / No	Data file	(txt):	Yes / No

Annex 2. Report

For both experiments:

1. Plot the pulse and response curves.
2. For the response curves:
    - Normalise the F-curves so that F(end) = 1, and
    - Get the E(t) values from the data

    Hint: Since the data is recorded discontinuously use numerical differentiation:

    $$\frac{\Delta\lambda}{\Delta t}$$

    With **Δt = 0.5 s** (for the "new" reactors) or **Δt = 1 s** (for the "old" CSTR), as your data was recorded with such frequency.

3. Compare the mean residence time, $\bar{t}$, with the space time, τ.
4. Calculate the variance, σ^2, and the standard deviation, σ.
5. The following information may be useful:

Feature	**CSTR (old version)**	**PFR (new version)**	**CSTR (new version)**
Volume (adjusted / fix)	890 mL (adjusted)	290 mL (fix)	290 mL (adjusted)
Volumetric flow (Pump power [%])	1.8 mL/s (50%)	2.60 mL/s (50%)	3.0 mL/s (50%)
Volumetric flow (Pump power [%])	2.6 mL/s (70%)	3.10 mL/s (70%)	3.4 mL/s (70%)
Volumetric flow (Pump power [%])	3.3 mL/s (90%)	3.35 mL/s (90%)	3.7 mL/s (90%)

6. We are aware that there is much space for improvement. **Give 3 examples of what could be improved, and suggest constructive solutions.**

The report and calculations must be done with the data that you generated in the practical training. If that is not the case, for any reason, the practical training will be graded as Failed.

The following documents must be submitted along with the report:

- Signed cover page
- Signed time log
- Working files used
    i. Excel file, <u>**OR**</u>
    ii. Matlab script and text file with the data
- 2x printed screenshots (one for each experiment)
- 2x pdf-prints from the Gunt Software (one for each experiment)
- An example of the last two can be found in the next page.

Keep your report concise (!), because for every graph, every table and at the end of every paragraph that you submit, it must be clearly stated:

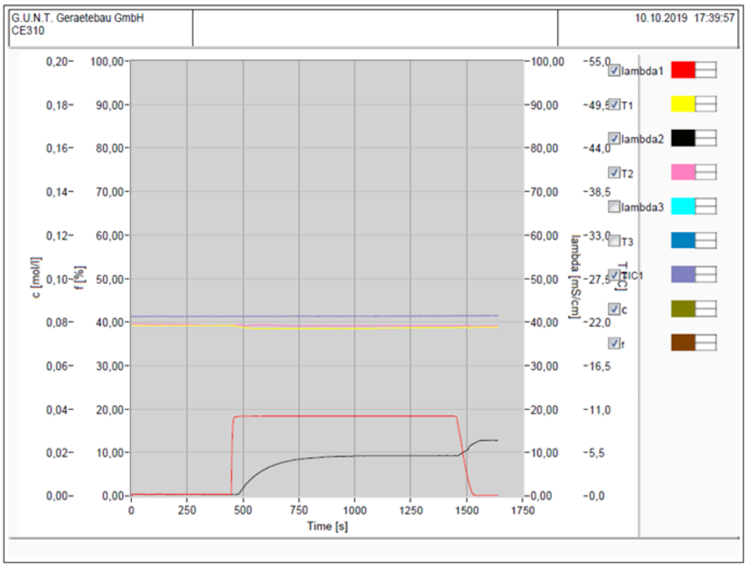
- **Who wrote/prepared it, and**
- **If it was modified, by whom and in which quality e.g. grammar, style correction, calculation, interpretation, etc.**

Furthermore, given prior experience, **<u>your report will be checked for plagiarism</u>**.

If a report or parts of it were plagiarised, there may be grave consequences for the ENTIRE GROUPS
(Those who shared AND those who received)

In case you require more information on these rules, refer to Prof. Dr.-Ing. Frank Platte, responsible for this Practical Training.

Example of a screenshot



Example of a Gunt-Software-generated graph

<u>Preparation Questions</u>

Safety & Preparation

1. List the different protective clothing that you must wear in a lab.

2. On top of a lab coat and the proper clothing and shoes, what other safety measures may be needed in a lab?

3. With which symbol are emergency exits marked? a)  b)  c) 

4. Name three pieces of the safety equipment available in the lab.

5. Where shall you work in the lab when toxic/hazardous fumes can occur?

6. Which documents must always be read beforehand *and* at hand when working in the lab?

7. Before you start with the experiment, you need to be familiar with the material safety data sheets of…

The Chemical Reactor System

8. A <u> (a) </u> is the place (vessel, tank, or pipe) in which the reaction takes place, while the <u> (b) </u> includes the machines, apparatuses, piping and fittings.

9. What is the purpose of a chemical reactor system?

10. Name 3 types of basic chemical reactors.

11. Give an example of a continuously operated chemical reactor.

12. Give an example of a discontinuously operated chemical reactor.

13. Give 2 examples of properties or conditions that can be controlled in a chemical reactor.

Process Flow Diagram & Equipment Operation

14. The ______ shows the general flow of processes and major equipment of a system.

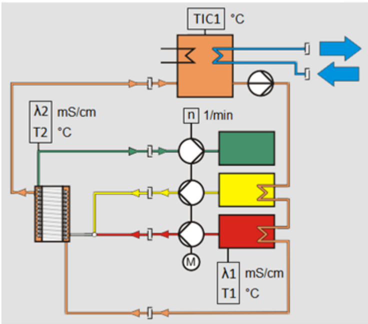
15. What does a Process Flow Diagram usually includes?

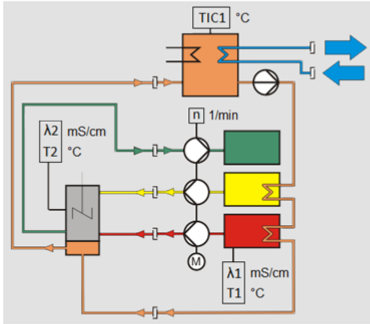
16. What does the Process Flow Diagram not show?

17. In a Process Flow Diagram, what do the symbols , ,  represent?

18. In a Process Flow Diagram, what do the symbols , ,  represent?

19. Assign the following process flow diagrams to the corresponding reactor system.

a) 

b) 

20. Where does the mixing of the reactants occur?
a) In the Reactants' tank   b) In the Reactor   c) In the Products' tank

21. What is the difference between a CST and a CSTR?

22. Name two types of pumps.

23. What piece of equipment do you need to use/power a pump?

24. What peculiarity of the "new" chemical reactor systems in our lab leads into serious setbacks in the operation of the system?

25. Which component of the cooling system let you know if there is enough cooling water available?

26. With the help of heat exchangers in the temperature control fluid's tank, the reactant tanks and the reactor itself can be:

Solutions, Reactants & Conductivity

27. What information is required to prepare a solution with a salt?

28. The preparation of 60mL NaCl solution (0.1M) can be done in a simple <u>dilution</u> step by mixing ____ of NaCl soln. (0.2M) with ____ of water.

29. What property is traced during the Residence Time Distribution practical training?

30. What is the conductivity value of air?

31. What is the conductivity value of deionized water?

32. What is the conductivity value of tap water?

33. Order of the following substances [Deionized water, NaCl 0.1M, NaCl 0.2M, tap water] from low to high conductivity.

34. What substance shall be used for rinsing the sensors?

35. What is the food colorant in our practical training used for?

36. Does the addition of food colorant lead to a significant change in the conductivity of tap water?

Hint: Compare the conductivity measurements for tap water with the conductivity measurement for the aqueous solution of the colorant. Keep in mind that small changes in measurement can be caused by the measurement inaccuracy of the sensors.

37. What happens if water is added to a tank that contains NaCl solution?

38. Name the reactants which are used for the Saponification reaction?

39. Why do you have to be very careful when working with the reactants needed for the Saponification practical training?

40. The electrical conductivity of sodium hydroxide (NaOH) is _____ compared to the conductivity of Ethyl acetate (EtOAc).

Residence Time Distribution

41. What does the residence time (also called age) indicate?

42. The concept of residence time originated in models of chemical reactors, but it can also be applied to other fields? Give two examples!

43. What do you need to measure the residence time distribution E(t) of a chemical reactor?

44. A _____ is a substance with specific properties that allow observing or identifying the behavior of a given physical or chemical process.

45. What properties shall the ideal tracer have?

46. In which categories can a tracer be classified <u>in terms of time</u>?

47. In which categories can a tracer be classified in terms of <u>influence on the flow</u>?

48. Name 2 examples of a tracer and describe the change it triggers!

49. What tracer do we use in the Residence Time Distribution practical training?

50. List the properties and devices that are controlled during the conduction of the Residence Time Distribution experiment.

51. Which are the two most commonly used input signals for measuring the Residence Time Distribution?

52. What is needed to conduct the Dirac Pulse Procedure?

53. Why is the Dirac pulse theoretically the ideal input signal for determining the Residence Time Distribution?

54. What kind of response curve is obtained from a (normalized) Step input signal?

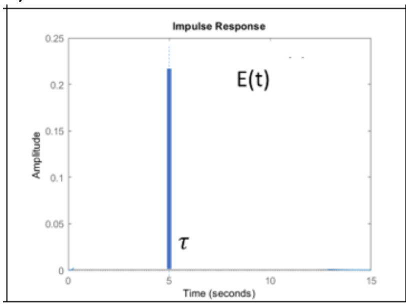
55. What is the independent variable (x-axis) in the Residence Time Distribution plot?

56. What is the dependent variable (y-axis) in the Residence Time Distribution plot?

57. Which effect does the perfect back-mixing of CSTRs (i.e. continuous washing out the traces) trigger?

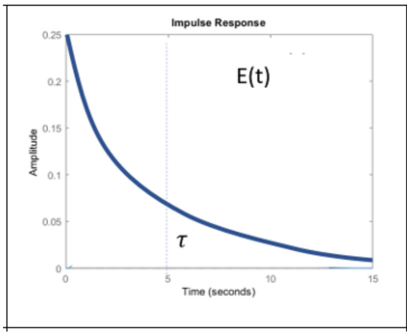
58. Assign the shown pulse responses to the proper ideal reactor.

a)



$$E(t) = \delta(t - \tau)$$

b)



$$E(t) = \frac{1}{\tau}\exp\left(-\frac{t}{\tau}\right)$$

59. Name 2 effects that lead to non-ideal behavior in real reactors?

60. In process engineering, why is it so important to analyze the RTD?

HOCHSCHULE RHEIN-WAAL Rhine-Waal University of Applied Sciences	**Operating Directive** Acc. § 5 Arbeitsschutzgesetz	Number: T-001-E-PBEa Page **1** of **2**
Faculty Life Sciences	**Working place/ Field of activity:** Laboratory 10 013	

Procedure of the Experiment

BE16 Practical Training A

Residence Time Distribution – Real Reactor Behavior

Hazards for humans and the environment

- Risk of noise-induced hearing loss:
  a) The reactors' sound levels are over 85 dB.
- Mechanical hazards:
  a) Long hair can get tangled in the rotating shafts of the stirring devices
  b) Moving parts in the old version reactor (2x small peristaltic pumps)
- Electrical hazards:
  a) If mishandled, liquids that get in touch with electricity-conducting cables
- Risk of burning:
  a) If mishandled (the practical training is to be conducted at room temperature), the reactors can be hot during the operation.
- Risk of being cut:
  a) If mishandled, glassware can shatter, and result in flesh wounds.

Safety measures and code of conduct

- Reading the present document and passing a pre-experiment test are required to conduct the experiment.
- Perform the experiment according to the instructions.
- Proceed in a proper, orderly way.
- Wear protective clothing, eye protection, and hear protection.
- Earplugs (37 dB noise reduction) are provided, must be used while the reactors are in operation, and their use must be controlled.
- Long hair is to be kept tied back and up, or kept inside cloths.
- Moving parts must not be touched.
- The reactors must be turned off and unplugged before cleaning them.
- Keep the device clean.
- Liquids are to be kept away from cables conducting electricity.
- Although protective gloves are not required, they are provided.

Conduct in emergencies | **Emergency call: 112**

- The reactor or the general electricity supply must be immediately turned off in case of:
  - Spillage, visible damages, unusual smells or sounds, formation of smoke, and hair entanglement on the rotor shaft
  - Apply a cause study to find the error. Do not proceed until it is safe to operate the equipment.
- In case of Fire/Emergency use the Emergency exits and proceed directly, in a calm and orderly fashion, to the Emergency meeting point outside Building 8. Report to your supervising instructor in this area before leaving the campus.
- All emergency exits and evacuation paths must remain free of obstacles!

HOCHSCHULE RHEIN-WAAL
Rhine-Waal University of Applied Sciences

Operating Directive

Acc. § 5 Arbeitsschutzgesetz

Number: T-001-E-PBEa

Page **1** of **2**

Faculty Life Sciences

Working place/ Field of activity: Laboratory 10 01 013

Procedure of the Experiment

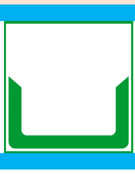
BE16 Practical Training A

Residence Time Distribution – Real Reactor Behavior

First aid | **Emergency call: 112**

- Take injured persons out of danger.
- Provide first aid, paying attention to self-protection.
- Call first aid (**112**) and within the Hochschule (-**299**).
- Cuts and scalps:
  a) Use disposable gloves and then a first-aid kit.
  b) Stop bleeding where possible.
  c) Apply dressings.
- Burns:
  a) Immediately remove clothing affected by hot substances.
  b) Submerge affected body parts in cold water or place under running cold water immediately, until pain relief sets in.
  c) Leave burns affecting face and eyes undressed.
- Do not leave injured people unattended until the rescue service arrives.

Proper disposal



- Water and sodium chloride solution (0.2 M) can be disposed into the sewage.