

Autumn Semester

Practical training on Residence Time Distribution



Table of Contents

1	Fundamentals	1
1.1	Introduction.....	1
1.2	Experimental methods.....	1
2	Residence time distribution of apparatuses	3
2.1	Mean residence time.....	3
2.2	Residence time distribution density function (pulse response).....	3
2.3	Residence time distribution in ideal apparatuses.....	4
2.3.1	Continuous stirred tank reactor (CSTR).....	4
2.3.2	Stirred tank cascade.....	5
2.3.3	Idealized pipe flow.....	6
3	Test plant	9
3.1	Setup.....	9
3.2	Technical data.....	10
4	Experimental procedure	10
5	Report	Error! Bookmark not defined.
6	Literature	14
7	Symbol table	14

1 Fundamentals

1.1 Introduction

The constant feeding of an apparatus and removal of products at the same time is characterizing a *continuous process*. The feed in general can be a multiphase mixture of gas, liquid and solid substances. The properties and the quality of the products strongly depend on the residence time of the different substances in the reactor. Thus, for the design of such continuously operating apparatuses, it is necessary to have proper information concerning the *residence time distribution*.

In case of a *discontinuously* operating reactor (batch), the residence time is given by the time between feeding and draining of the system. However, in continuously operated apparatuses, the molecules of the reacting substances don't generally remain in the apparatus for the same time length. Part of the substances leaves the apparatus earlier in comparison to another fraction, which stays longer. The residence time can therefore no longer be described by a single value, but rather by a timely distribution. This mathematical function is called *residence time distribution* (dt.: *Verweilzeitverteilung*). The theoretical investigation of this distribution is often not accurate, because several factors have to be considered, for example the geometry, the operating conditions and the material properties, which are often not constant in the system (p, T). Consequently, the residence time distribution is often determined by experiment.

1.2 Experimental methods

The experimental investigation of the residence time distribution in a streamed apparatus is generally based on a property modification of the fluid flow at the inlet, which can be detected and measured at the outlet of the system.

In many cases the modified property is the concentration of a non reactive substance in the feed flow. This substance is named *tracing substance* or shortly *tracer* and should fulfill the following criteria:

- simply and accurately detectable
- similar fluid properties as the main fluid
- no interactions with walls and interfaces
- no reactions with other components

In principle, three different types of tracer signals are used at the inlet:

- **Pulse:**
Addition of a small amount of tracer in a preferably short period of time. The amplitude of the idealized signal peak is infinite and the time extension is infinitesimal (*Dirac pulse* or δ -pulse). In the laboratory such a pulse has rather

the shape of a normal probability curve (Gauss). It is important for the initial signal that the pulse is nearly symmetric and that it has a narrow base width (time extension) compared to the response signal at the outlet of the apparatus.

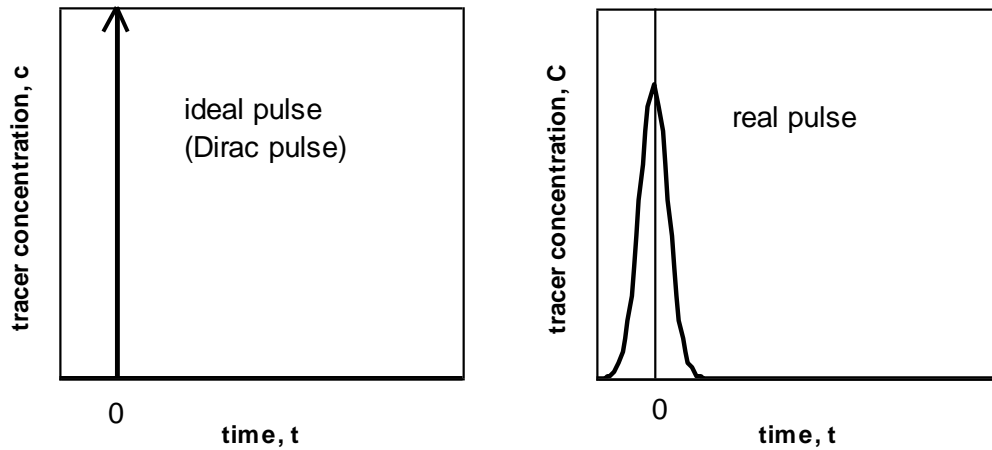


Figure 1: Comparison of an ideal pulse (left) and a pulse in reality (right)

- **Step:**
Cascaded change of the inlet concentration of a component
- **Sinus:**
Sinusoidal change of the concentration of a component by periodic stimulation at a certain position

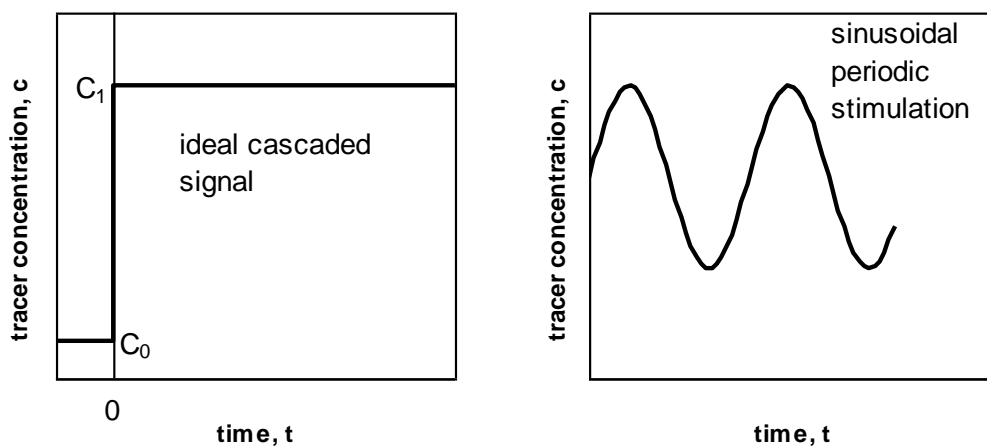


Figure 2: Cascaded signal and sinusoidal periodic stimulation of the concentration of a component

2 Residence time distribution of apparatuses

2.1 Mean residence time

The *mean or hydraulic residence time* is defined as the quotient of the streamed volume V_R and the volume flow rate \dot{V} .

$$\tau = \frac{V_R}{\dot{V}} \quad [s] \quad (2.1)$$

It can either be determined based on time averaging of the pulse response $C(t)$ at the outlet of the apparatus or simply by means of the residence time distribution density $E(t)$ (Section 2.2).

$$\tau = \frac{\int_0^{\infty} C(t) \cdot t dt}{\int_0^{\infty} C(t) dt} = \int_0^{\infty} E(t) \cdot t dt \quad (2.2)$$

By using the mean residence time, the residence time distribution function can be formulated dimensionless. Thus, it is possible to compare the residence time distribution of different systems to each other. The dimensionless time is mathematically formulated as the current time divided by the mean residence time.

$$\theta = \frac{t}{\tau} \quad [-] \quad (2.3)$$

2.2 Residence time distribution density function (pulse response)

The *residence time distribution density* $E(t)$ can be calculated by the normalization of the pulse response at the outlet $C(t)$ with respect to the whole amount of tracer used.

$$E(t) = \frac{C(t)}{\int_0^{\infty} C(t) dt} \quad [s^{-1}] \quad (2.4)$$

The dimensionless formulation of the residence time distribution density is obtained by a multiplication with the mean residence time τ .

$$E(\theta) = \frac{C(\theta)}{\int_0^{\infty} C(\theta) d\theta} = \tau \cdot E(t) \quad [-] \quad (2.5)$$

The area under the residence time distribution density function always equals 1 as a result of the normalization (2.4).

$$\int_0^{\infty} E(t) dt = \int_0^{\infty} E(\theta) d\theta = 1 \quad (2.6)$$

2.3 Residence time distribution in ideal apparatuses

2.3.1 Continuous stirred tank reactor (CSTR)

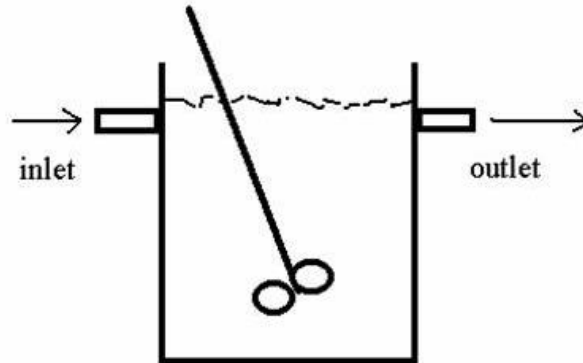


Figure 3: Ideal CSTR

An ideal mixed CSTR can be easily modeled by performing a mass balance of the injected tracer component. The tracer concentration at the outlet of the reactor is calculated as a response of the cascaded increase at the inlet ($C(t < 0) = 0$ to $C(t \geq 0) = C_{in}$). The assumption of ideal mixing means that the tracer concentration in the CSTR is equal to the outlet concentration.

The mass balance for the tracer can be formulated as:

$$C_{in} \cdot \dot{V} - C \cdot \dot{V} = V_R \frac{dC}{dt} \quad (2.7)$$

With the initial conditions mentioned above, the variables can be separated as follows:

$$-\int_0^t \frac{dt}{\tau} = \int_0^C \frac{dC}{C - C_{in}} \quad (2.8)$$

$$-\frac{t}{\tau} = \ln\left(\frac{C_{in} - C}{C_{in}}\right) \quad (2.9)$$

$$C(t) = C_{in} \exp(-t / \tau) \quad (2.10)$$

The residence time distribution density can now be obtained:

$$E(t) = \frac{C(t)}{\int_0^{\infty} C(t) dt} = \frac{1}{\tau} e^{-\frac{t}{\tau}} \quad (2.11)$$

In dimensionless form:

$$E(\theta) = e^{-\theta} \quad (2.12)$$

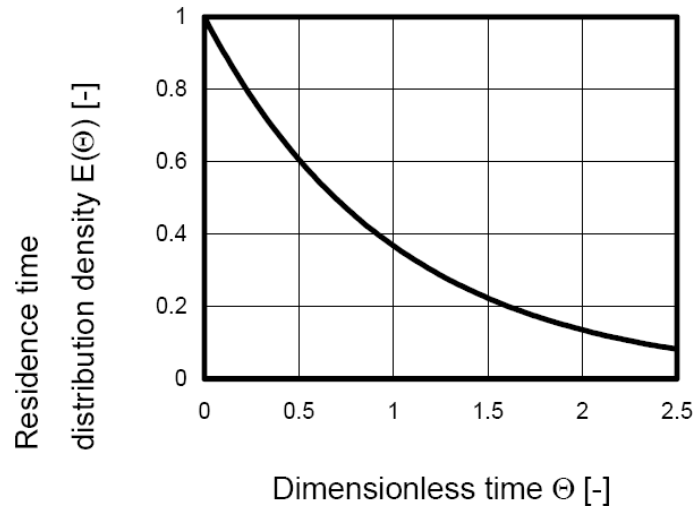


Figure 4: Residence time distribution density for an ideal mixed CSTR

2.3.2 Stirred tank cascade

A stirred tank cascade consists of several CSTRs which are connected with each other in series. The following equation for $E(t)$ is valid for a cascade consisting of n CSTRs with the same volume:

$$E(t) = \frac{1}{(n-1)!} \cdot \frac{1}{\tau} \cdot \left(\frac{t}{\tau}\right)^{n-1} \cdot e^{-\frac{t}{\tau}} \quad (2.13)$$

Formulated dimensionless:

$$E(\theta) = \frac{\theta^{n-1}}{(n-1)!} \cdot e^{-\theta} \quad (2.14)$$

where τ is the mean residence time in every single CSTR.

Figure 5 depicts the residence time distribution density for a variable number of single CSTRs. It can be noticed that, with increasing number of CSTRs, the residence time distribution density of a cascade approaches the residence time distribution density of an idealized pipe flow.

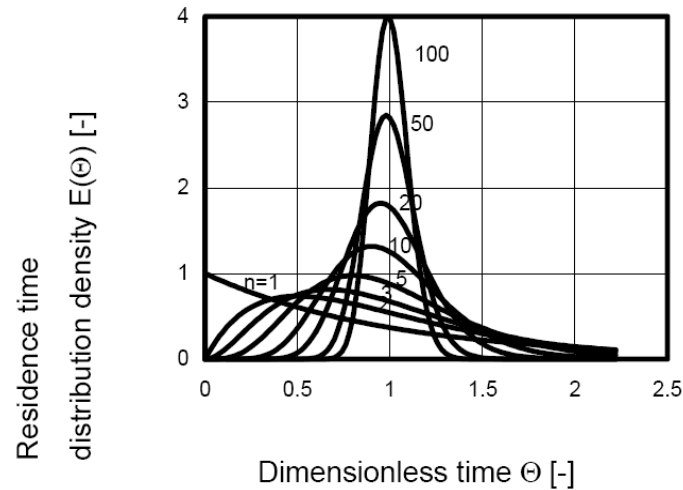


Figure 5: Residence time distribution densities for stirred tank cascades with different numbers of single CSTRs

2.3.3 Idealized pipe flow

A *frictionless, idealized pipe flow*, with a homogeneous flow velocity over the pipe cross section, leads to a flow pattern without axial mixing.

Any signal (pulse) at the inlet of this pipe can be detected unchanged and with a time delay at the outlet.

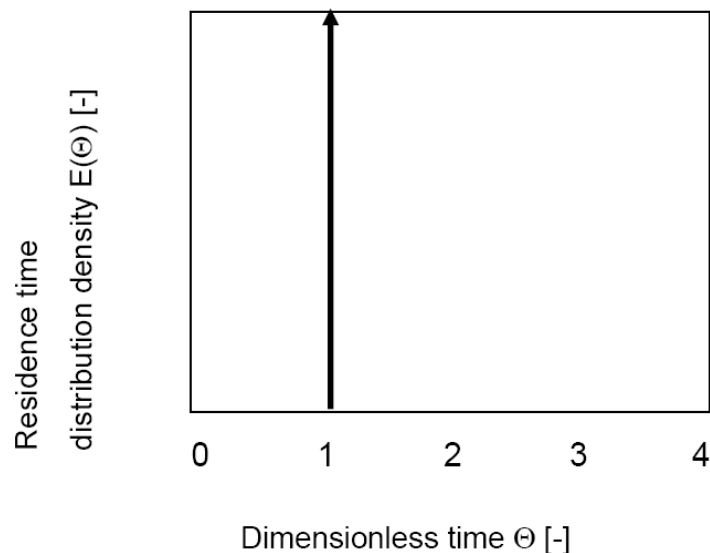


Figure 6: Residence time distribution density versus dimensionless time for pipe flow without backflow

The flow pattern mentioned above is usually called *plug flow* (dt.: *Pfropfen- oder Kolbenströmung*). The time delay of the signal at the outlet is equal to the residence time of the substances in the pipe and it is defined as:

$$\tau = \frac{V_R}{\dot{V}} = \frac{A \cdot L}{A \cdot u} = \frac{L}{u} \quad (2.15)$$

Such a plug flow can only be achieved with high flow velocities. In this case the turbulence is the dominant mechanism and therefore radial velocity gradients are compensated.

If the flow of a *Newtonian fluid* through a pipe is *laminar*, a parabolic velocity profile as a result of wall friction effects will be developed.

$$u(r) = u_0 \left[1 - \left(\frac{r}{R} \right)^2 \right] \quad (\text{for } 0 \leq r \leq R) \quad (2.16)$$

In equation 2.10, u_0 is the velocity on the axis of the pipe ($r=0$), which is twice than the average velocity \bar{u} .

$$\bar{u} = \frac{u_0}{2} \quad (2.17)$$

The mean residence time can be calculated as follows:

$$\tau = \frac{V_R}{\dot{V}} = \frac{A \cdot L}{A \cdot \bar{u}} = \frac{L}{\bar{u}} = \frac{2 \cdot L}{u_0} \quad (2.18)$$

The integration over the pipe cross section results in the following expression for the residence time distribution density in the laminar case:

$$E(t) = \begin{cases} 0 & \text{for } t < \frac{\tau}{2} \\ \frac{1}{2} \frac{\tau^2}{t^3} & \text{for } t \geq \frac{\tau}{2} \end{cases} \quad (2.19)$$

Formulated dimensionless:

$$E(\theta) = \begin{cases} 0 & \text{for } \theta < \frac{1}{2} \\ \frac{1}{2 \cdot \theta^3} & \text{for } \theta \geq \frac{1}{2} \end{cases} \quad (2.20)$$

Figure 7 shows that the residence time distribution density for laminar flows is very un-symmetrical. This means that in fact 80% of the fluid has a residence time in between $\tau/2$ and τ , but a significant amount of substance (about 5%) spends more than twice the time in the system. Since this effect is rarely desired, turbulent flow is generally preferred in order to achieve a narrower residence time distribution.

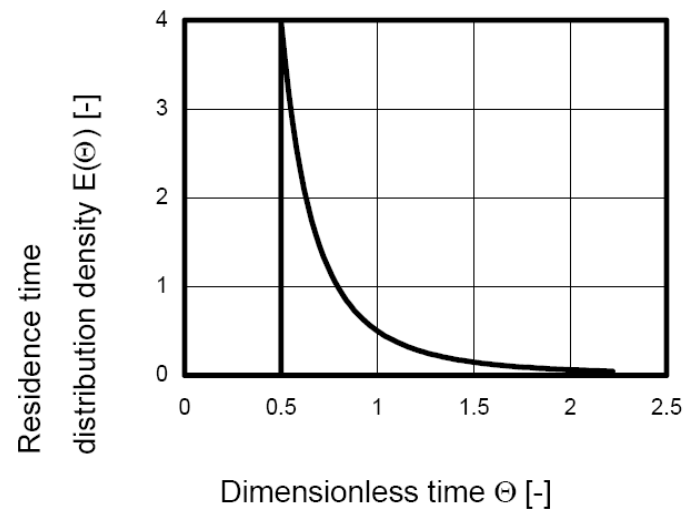


Figure 7: Residence time distribution density function for laminar pipe flow

3 Test plant

3.1 Setup

The laboratory course is held at a test plant, which consists of three CSTRs connected in series, a flow pipe and a packed column (Figure 9). The plant components are made of glass.

A concentrated KCL-solution is used as tracer. This tracer solution is injected with a syringe through a septum directly into the flow at predefined spots of the plant. At the same time the outlet concentration profile is measured by using a conductivity sensor. The base width of the initial pulse will significantly influence the investigation of the residence time distribution in the flow pipe. Therefore a concentration measurement is installed directly after the injection point of the tracer to verify the shape of the initial pulse.

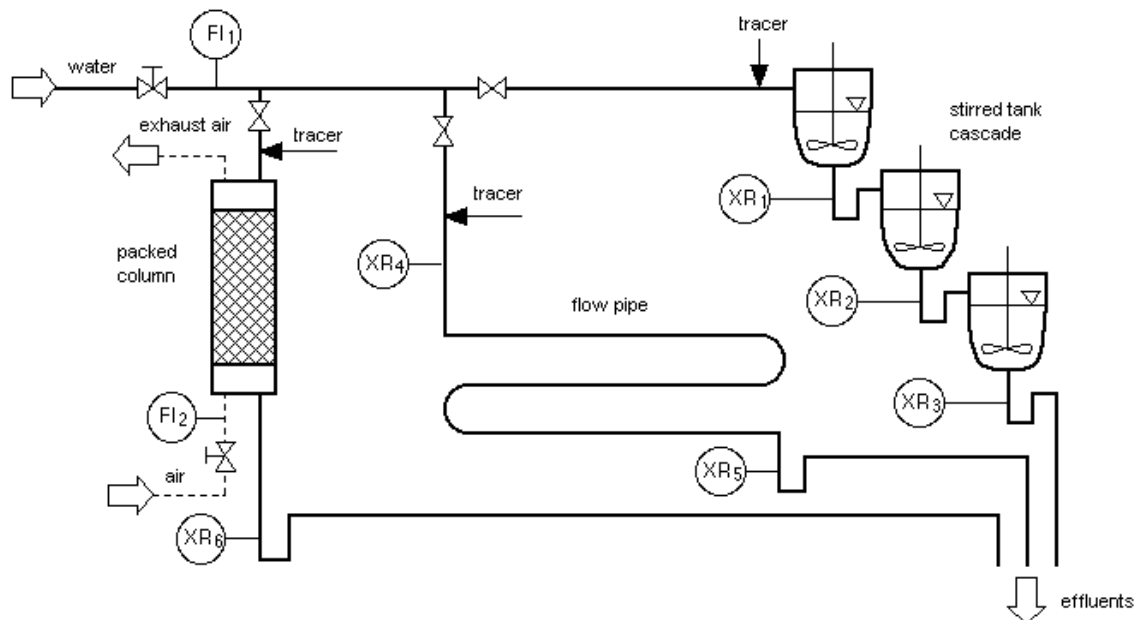


Figure 8: Flow chart of the test plant

The determination of the concentration by conductivity sensors was chosen because changes in concentration of soluble salts are fast and accurately detectable by this method. The sensors are inserted in the flow and an alternating voltage with known amplitude is applied. The electrical current between the two electrodes of every sensor is measured. The conductivity of the fluid depends linearly from the KCL-Ion concentration in it. Since the current is proportional to the conductivity [$1/\Omega$] of the fluid, the KCL-Ion concentration at the electrodes can be determined.

The concentrations are recorded at equal, adjustable time steps. The data files are saved on the computer in tabular form, so that they can be transferred to other computers for evaluation reasons.

3.2 Technical data

Stirred tank cascade			
Number of stirred tanks	n	3	-
Tank volume	V_{tot}	4	L
Filling level during operation*	V_{exp}	$\sim 3^*$	L
Water flow	\dot{V}	0-100	L/h
Flow pipe			
Inner diameter	D	15	mm
Length	L	2635	mm
Water flow	\dot{V}	0-370	L/h

* Exact filling level during operation should be determined during the experiment

4 Experimental procedure

In the laboratory course “residence time distribution” the following experiments are executed:

	Experiment	\dot{V}_{Water}	\dot{V}_{air}	Residence time model for evaluation
1	Stirred tank cascade	60 L/h	--	Stirred tank model
2	Flow pipe	60 L/h	--	Laminar flow
3	Flow pipe	360 L/h	--	Stirred tank & Dispersion model

Experiment 1: “Stirred tank cascade”

1. Close all valves.
2. Open the valve that leads to the CSTR cascade.
3. Carefully set the volumetric flow of water (see rotameter) to 60 L/h. Please do not go beyond 60 L/h as the syphons are not designed for higher flow rates. Usually it takes time for the flow to stabilize; therefore, remember to continuously regulate the volumetric flow during the course of the experiment.
4. Switch on the compressed air motors that actuate the stirrers.
5. Wait until all three tanks reach a steady water level.
6. Open the software LabView: Set the «Experiment» to Stirred Tank Cascade from the menu.
7. In LabView: “Experiment“ auf Stirred Tank Cascade umstellen;
 - Number of Scans: 20
 - Scans per Second: 20
8. Confirm your input by clicking on the green button.
9. Turn the conductivity probes such that the electrodes are parallel to the direction of the flow.
10. Please check that there are no air bubbles in proximity of the conductivity probes.
11. Zero the signal by clicking on “Concentration Offset”.
12. Fill the syringe with **5 mL** of tracer.
13. Pierce the membrane and rotate the needle such that the bevel is towards the direction of the flow. **Do not start the injection yet.**
14. Ideally, two people should carry out this step. Inject the tracer and simultaneously start recording the data by clicking on the green button “START”. The tracer should be injected as rapidly as possible (ideally as a Dirac pulse injection).
15. The measurement should run for approximately 20 to 30 minutes until the whole amount of tracer is flowed through the CSTR cascade.
16. Terminate the experiment by clicking on the red button “STOP”.
17. Save the measured data.
18. Export the data as txt-files for the analysis of the results.
19. Read out the water level in each of the three stirred tanks. To do so, stop stirring for a very short time.

V₁= _____ mL

V₂= _____ mL

V₃= _____ mL

Experiment 2: “Flow pipe 60 L/h”

1. Close all valves.
2. Open the valve to direct the flow to the flow pipe.
3. Flush the flow pipe extensively in order to eliminate air bubbles.
4. Set the water volume on the rotameter to 60 L/h.
5. In LabView: Set “Experiment” on Flow Pipe
 - Number of Scans: 20
 - Scans per Second: 100
6. Confirm your input by clicking on the green button.
7. Turn the conductivity probes such that the electrodes are parallel to the direction of the flow.
8. Please check that there are no air bubbles in proximity of the conductivity probes.
9. Zero the signal by clicking on “Concentration Offset”.
10. Fill the syringe with **1 mL** of tracer.
11. Pierce the membrane and rotate the needle such that the bevel is towards the direction of the flow. **Do not start the injection yet.**
12. Ideally, two people should carry out this step. Inject the tracer and simultaneously start recording the data by clicking on the green button “START”. The tracer should be injected as rapidly as possible (ideally as a Dirac pulse injection).
13. The measurement should run for approximately 1-2 minutes until the whole amount of tracer flows through the flow pipe.
14. Terminate the experiment by clicking on the red button “STOP”.
15. Save the measured data.
16. Export the data as txt-files for the analysis of the results.

Experiment 3: “Flow pipe 360 L/h”

1. Fill the syringe with **1 mL** of tracer.
2. Set the water volume on the rotameter to 360 L/h.
3. Pierce the membrane and rotate the needle such that the bevel is towards the direction of the flow. **Do not start the injection yet.**
4. Ideally, two people should carry out this step. Inject the tracer and simultaneously start recording the data by clicking on the green button “START”. The tracer should be injected as rapidly as possible (ideally as a Dirac pulse injection).
5. The measurement should run for approximately 1-2 minutes until the whole amount of tracer is flows through the flow pipe.
6. Terminate the experiment by clicking on the red button “STOP”.
7. Save the measured data.
8. Export the data as txt-files for the analysis of the results.

End:

1. Clean and flush the plant with water and compressed air.
2. Empty the plant with the help of compressed air.
3. Shut down computer, frequency generator and „Blue Box“

5 Literature

P. Grassmann: Physikalische Grundlagen der Verfahrenstechnik, 3. Auflage, Sauerländer, Aarau 1982

C. G. Hill: An Introduction to Chemical Engineering Kinetics & Reactor Design, Wiley, New York 1977

6 Symbol table

Variables

A	[m ²]	Cross section surface
Bo	[-]	Bodenstein number
C, C_o, C_1	[mg/ml]	Tracer concentration
d	[m]	Diameter of the Raschig rings
D	[m]	Diameter
D_{eff}	[m ² /s]	Dispersion coefficient
E	[s ⁻¹]	Residence time distribution density
F	[-]	Transfer function
H	[m]	Height of the packed column
L	[m]	Length
n	[-]	Number of stirred tanks
Pe	[-]	Peclet-number
r	[m]	Distance to the pipe axis
R	[m]	Radius of the pipe
t	[s]	Time
u	[m/s]	Velocity
\bar{u}	[m/s]	Mean velocity
u_o	[m/s]	Velocity in the pipe axis
\dot{V}	[m ³ /s]	Volume flow rate
V_R	[m ³]	Streamed volume
x	[-]	Variable
y	[-]	Variable
Z	[m]	Coordinate
θ	[-]	Dimensionless time
σ^2	[s ²]	Variance

τ	[s]	Mean residence time
Ω	[Ω]	Resistance

Indices

<i>aus</i>	out
<i>ein</i>	in
<i>ges</i>	total
<i>i</i>	control variable
<i>j</i>	control variable
<i>Luft</i>	air
<i>Sprung</i>	jump
<i>Wasser</i>	water
1	begin
2	end