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**Measurements of residence time distribution in  
columns with different fillings**

**Practical course**

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# 1 Theory

When designing both continuous and batch processes, it is necessary to fully understand the Residence Time (*Verweilzeit*) for the substances flowing through the process, more specifically through the reactor (*Reaktor*). This time delineates the quality of the products leaving the process. Not enough time could lead to very little product and much un-reacted reactants, whereas too much time could lead to secondary or tertiary reactions occurring that form products that are unusable. By measuring the Residence Time at different process conditions, the process can be optimized for the results, products that wish to be obtained.

There are profound differences between continuous and batch processes. However, since this experiment dealt with a continuous process, the rest of this paper will focus on such. In a continuous process, a single Residence Time cannot be measured. This is due to the fact that molecules entering the reactor do not leave the reactor at the same time as one another. There is a distribution with respect to time that they leave. Theoretically, it seems that this cannot be the case that they would leave at the same time and there would be a single value for the Residence Time. However, this is not the fact due to such things like geometry of the reactor (process), concentration gradients, process conditions, etc. It is these differences that cause there to be a distribution, and this fact leads to the reason why in continuous processes a Residence Time Distribution (RTD) (*Verweilzeitverteilungen*) is measured instead of a single Residence Time value.

## 1.1 Residence Time Distribution

The **residence time distribution** (RTD) of a chemical reactor is a probability distribution function that describes the amount of time that a fluid element could spend inside the reactor. 'Fluid element' is a small volume of fluid where continuous properties, such as concentration, can still be defined. The RTD function, external residence time distribution,  $E(t)$ , measures the time that the various fractions of 'fluid element' reside in the reactor (macromixing); it gives no information about the mixing details at a molecular level (micromixing).

The expressions of the RTD functions can be derived for the ideal reactors, as well as for any association of PFR and CSTR elements. Chemical Engineers employ the RTD to characterize the mixing and flow within reactors and to compare the behavior of real reactors to their ideal

models. This is useful, not only for trouble shooting existing reactors, but in estimating the yield of a given reaction and designing future reactors.

The theory of residence time distributions generally begins with three assumptions:

1. The reactor is at steady-state,
2. Transports at the inlet and the outlet takes place only by advection, and
3. The fluid is incompressible ( $v = \text{constant}$ ).

## 1.2 Determining the RTD Experimentally

Residence time distributions are measured by introducing a non-reactive tracer into the system at the inlet. The concentration of the tracer is changed according to a known function and the response is found by measuring the concentration of the tracer at the outlet.

The selected tracer should not modify the physical characteristics of the fluid (equal density, equal viscosity) and the introduction of the tracer should not modify the hydrodynamic conditions. A suitable tracer material for RTD-experiments should exhibit similar physical properties, *i.e.* viscosity, diffusion coefficient, density, to the flow medium and in particular the materials used should behave inertly, not adhere or adsorb on to the reactor walls and be easily detectable. In particular this last aspect is of critical importance for the design of an optimal system to measure residence time distributions in microreactors. The experimental determination of the residence time distribution requires a well-defined introduction of a tracer species into the reactor inlet and the detection of its transient concentration leaving the reactor outlet. In general, the change in tracer concentration will either be a *pulse* or a *step input*.

## 1.3 Pulse Input Experiment

In a pulse input, an amount of tracer  $N_0$  is suddenly injected in one shot into the feedstream entering the reactor in as short a time as possible. The outlet concentration is then measured as a function of time. Typical concentration– time curves at the inlet and outlet of an arbitrary reactor are shown in Figure 1.

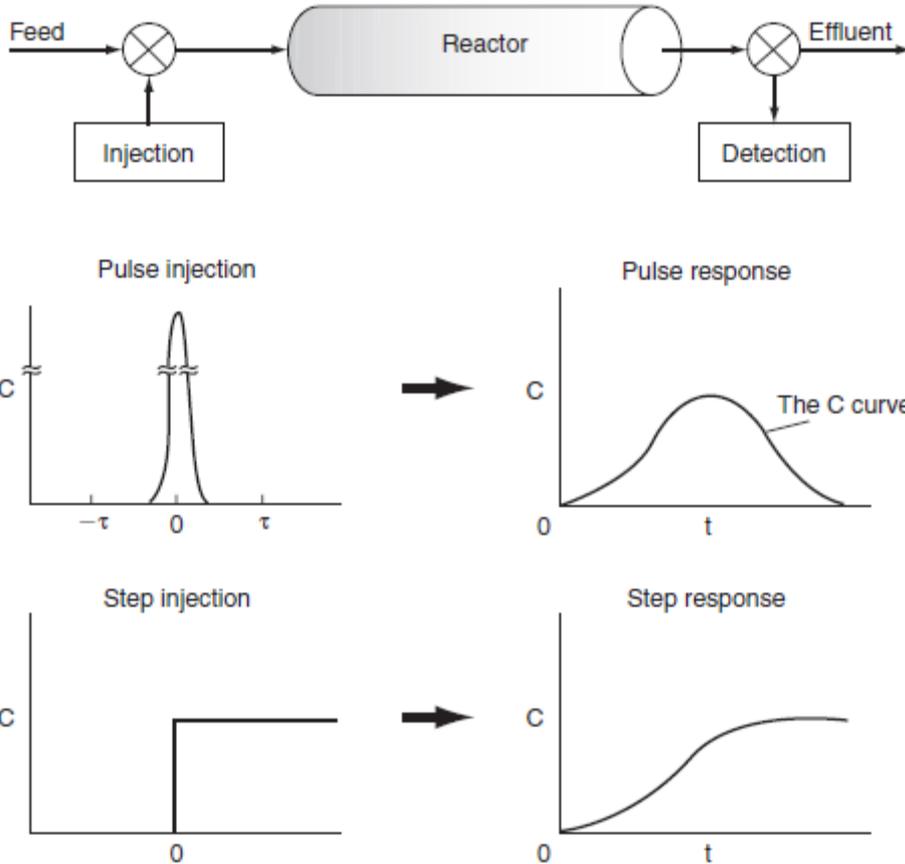


Figure 1: RTD measurements

The effluent concentration–time curve is referred to as the  $C$  curve in RTD analysis. We shall analyze the injection of a tracer pulse for a single-input and single-output system in which *only flow* (i.e., no dispersion) carries the tracer material across system boundaries. First, we choose an increment of time  $\Delta t$  sufficiently small that the concentration of tracer,  $C(t)$ , exiting between time  $t$  and  $t+\Delta t$  is essentially the same. The amount of tracer material,  $\Delta N$ , leaving the reactor between time  $t$  and  $t+\Delta t$  is then:

$$\Delta N = c(t)v\Delta t \quad \text{eq. 1}$$

where  $v$  is the effluent volumetric flow rate. In other words,  $\Delta N$  is the amount of material exiting the reactor that has spent an amount of time between  $t$  and  $t+\Delta t$  in the reactor. If we now divide by the total amount of material that was injected into the reactor,  $N_0$ , we obtain

$$\frac{\Delta N}{N_0} = \frac{vc(t)}{N_0} \Delta t \quad \text{eq. 2}$$

which represents the fraction of material that has a residence time in the reactor between time  $t$  and  $t+\Delta t$ .

For pulse injection we define

$$E(t) = \frac{vc(t)}{N_0} \quad \text{eq. 3}$$

so that

$$\frac{\Delta N}{N_0} = E(t)\Delta t \quad \text{eq. 4}$$

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If  $N_0$  is not known directly, it can be obtained from the outlet concentration measurements by

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$$\frac{dN}{dt} = c(t)v \quad \text{eq. 5}$$

and then integrating, we obtain

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$$N_0 = \int_0^\infty vc(t)dt \quad \text{eq. 6}$$

The volumetric flow rate is usually constant, so we can define  $E(t)$  as

$$E(t) = \frac{c(t)}{\int_0^\infty c(t)dt} \quad \text{eq. 7}$$

The integral in the denominator is the area under the  $C$  curve. An alternative way of interpreting the residence-time function is in its integral form:

$$\int_{t_1}^{t_2} E(t)dt = \text{Fraction of material leaving the reactor that has resided in the reactor for times between } t_1 \text{ and } t_2 \quad \text{eq. 8}$$

We know that the fraction of all the material that has resided for a time  $t$  in the reactor between  $t=0$  and  $t=\infty$  is 1; therefore,

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$$\int_0^{\infty} E(t) dt = 1 \quad \text{eq. 9}$$

Analogously, we have

$$\int_t^{\infty} E(t) dt = 1 - F(t) = \boxed{\text{Fraction of effluent that has been in reactor for longer than time } t} \quad \text{eq. 10}$$

The  $F$  curve is another function that has been defined as the normalized response to a

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The average residence time is given by the first moment of the age distribution:

$$t_m = \frac{\int_t^{\infty} tE(t) dt}{\int_t^{\infty} E(t) dt} = \int_t^{\infty} tE(t) dt \quad \text{eq. 11}$$

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If there are no dead, or stagnant, zones within the reactor, then  $t_m$  will be equal to  $\tau$ , the residence time calculated from the total reactor volume and the volumetric flow rate of the fluid:

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$$\tau = \frac{V}{v} = t_m \quad \text{eq. 12}$$

The higher order central moments can provide significant information about the behavior of the function  $E(t)$ . For example, the second central moment indicates the variance ( $\sigma^2$ ), the degree of dispersion around the mean.

$$\sigma^2 = \int_0^{\infty} (t - t_m)^2 E(t) dt \quad \text{eq. 13}$$

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## 2 Experiment

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The theoretical value or curve for this Residence Time Distribution is extremely difficult to calculate and this is why more often it is found experimentally. The reason that it is hard to determine is because it is usually very inaccurate since many factors involving the system, components, and process must be considered in the calculation. Some of these factors were

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also mentioned above when discussing why the Residence Time is not a single value. For this reason, RTD is more often determined through experimental means by using a tracer (Indikator) injected into the process and measuring this tracer in the outlet stream with respect to time. The tracer can be measured through changes in conductivity or concentration; however in this experiment, the conductivity of the exit stream was measured. The objective of this experiment is to find an average residence time (distribution) for the process of columns with different packings.

## 2.1 Experimental Procedure (*Versuchsdurchführung*):

The experiments performed used a simple salt solution 3M NaCl, as the tracer. The tracers were added as a “pulse” (*Puls*) to the overall process. “Pulse” can be defined as being a small amount of the tracer injected in a single shot over a short time period into the feed stream of the reactor. 1 mL of the 3M NaCl solution has to be injected in the plant for each trial in this experiment with an overall flow rate of 300 L/hr.

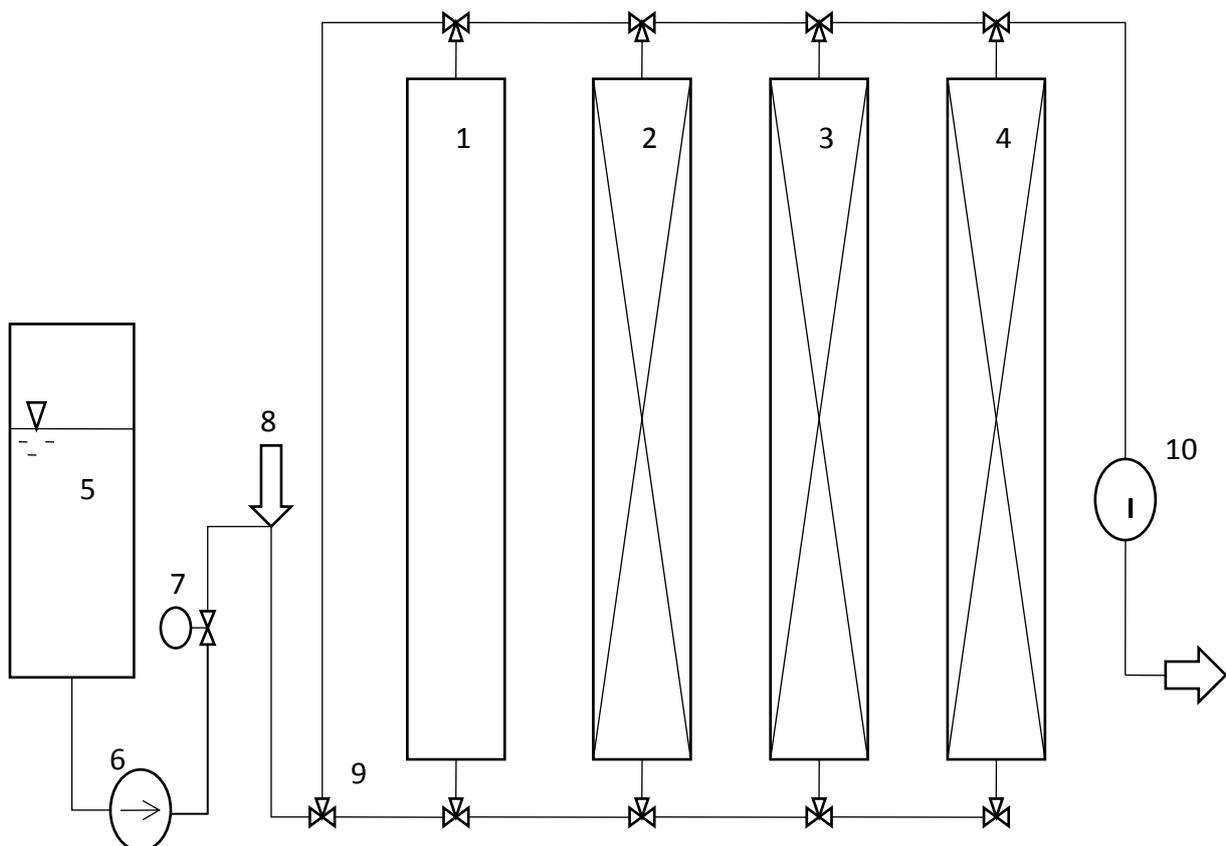


Figure 2: *Experimental apparatus*

Glaskugeln mit 1 mm Durchmesser
Glaskugeln mit 10 mm Durchmesser
unregelmäßige Schüttung aus gleich gefertigten Füllkörpern

1. Empty column
2. Column with glas packing d1mm
3. Column with glas packing d10mm
4. Column (defect)
5. Vessel for Deionized water
6. Pump
7. Rotameter
8. Syringe (injection of the NaCL solution)
9. Tree way valves
10. Conductivity device

Figure 2 depicts the experimental apparatus used to calculate the RTD curves for the different tracers. Deionized water is pumped through a series of tubes to the reactor, where it passes through and then is emptied to a drain. The figure shows where the tracer is injected using a syringe as well as where the conductivity of the outlet stream is measured as a function of time. The sensor used in the process apparatus measures the conductivity in microSiemens ( $\mu\text{S}$ ) and this is read by the transducer which then outputs it to the data logger and ultimately to the computer.

It is important to note that the picture depicts a series of simulated reactors labeled as 1-4. However, we will only use 3 of these reactors for the experiment. The experiment has to be run such that the process reaches steady state before inserting the tracer. Once steady state is achieved, the data logger has to be run, and the tracer has to be injected. The experiment will be stopped, when the tracer is no longer apparent in the outlet stream. The data has to be saved to an Excel file for future analysis.

### **3 Tasks**

1. Obtain the residence time distribution of the tubular reactor (pulse signal)
2. Determine the  $E(t)$ -curve
3. Determine the  $F(t)$ -curve
4. Calculate the average residence time (the first moment of the distribution)
5. Compare the results of  $E(t)$ ,  $F(t)$  and  $\tau$  using graphical plots

#### **References:**

**Levenspiel O. (1999): Chemical Reaction Engineering, John Wiley & Sons, Inc. Third Edition**

**Pereira C.J, Leib T. M. (2008): Perry's chemical engineers' handbook, Section 19: Reactors, McGraw-Hill eBooks**