Practical Exercises in Physical Chemistry

Advanced Level

Freie Universität Berlin, Institut für Chemie und Biochemie

Block III: Kinetic

Stopped-flow

How to operate the stopped-flow setup:

A part of the experimental setup is made of glass and is extremely fragile. Especially, the syringes should not be tilted. Never use force on the syringes and the three-way cocks.

a) Set the switch “supply” in the position “OFF”, turn the button “bias” to the left stop, set the switch “equipment” in the position “ON”.

b) Switch on the oscilloscope and set the sensitivity to 1 V/cm and select “external trigger”. Switch on the lamp of the photometer unit using the switch “supply”. Tune the button “bias” to locate the signal in the low middle of the oscilloscope screen. Press the key “arm” to activate the storage mode of the oscilloscope.

c) The experimental setup used in this work is shown in Fig. 1. Fill the reservoirs with distilled water. Turn the cocks 1 and 2 to connect the reservoirs with the syringes 1 and 2. Slowly pull the driving unit back to fill the syringes with water. You may need to move the driving unit back and forth to remove air bubles. Turn the cock 3 to connect the flow cell, which is inside the photometer unit, with the syringe 3. Turn the cocks 1 and 2 to connect the syringes 1 and 2 to the flow cell. Push the driving unit until the syringe 3 hits the microswitch. Check whether the trigger switched on the storage oscilloscope. Turn the cock 3 to connect the syringe 3 to the outlet. Check if the outlet pipe is inserted into the waste bottle. Empty the syringe 3 gently. Turn the cocks 1 and 2 to connect the reservoirs with the syringes 1 and 2. Turn the cock 3 back to connect the flow cell with the syringe 3. Repeat the whole set several times to wash the system and become familiar with the

setup. Be careful not to suck in the fraction from the flow cell into the syringes 1 and 2!

d) Prepare solutions whose compositions are given in the following table. The solutions X have to be filled up with water to 50 ml and the solution Y to 100 ml in graded flasks.

e) Make the reservoirs and the syringes 1 and 2 free of water. Fill the reservoir 1 with the solution X1 and the reservoir 2 with the solution Y. Repeat the set described above two times to remove distilled water from the system.

f) Set the sensitivity to 50 mV/cm and tune the button “bias” to locate the signal in the low middle of the oscilloscope screen. Set the time scale of the oscilloscope in the range 20-100 ms/cm. Press the key “arm” to activate the storage mode of the oscilloscope.

g) Push the driving unit until the syringe 3 hits the microswitch. The oscilloscope should now record the curve similar to one shown in Fig. 2. Take care that you use the full size of the screen and that you can actually see the equilibrium value U_∞ on the screen!

h) Switch on the plotter and plot the cure by pressing the button “manual plot” at the oscilloscope. Write the identification number of the solution and the time deviation rate on the plotting sheets. Repeat the experiment at least two times.

i) Make the reservoir 1 and the syringe 1 free of the solution X1. Fill them with the solution X2. Rinse the system with the solutions X2 and Y as described above and make your experiments further.

j) After the final experiment with the solution X5 rinse the system with distilled water.

k) Clean up the working place at the end. Switch off the experimental set-up.

Substance	Concentration	Amount in solution L						Unit
		X1	X2	X3	X4	X5	Y	
$FeCl_3$ in 0.2 M $HCLO_4$	0.1 M	5	5	5	5	5	0	ml
KSCN	0.01 M	0	0	0	0	0	10	ml
$HClO_4$	1.0 M	0	1	2	5	10	0	ml
$NaClO_4$	1.0 M	19	18	17	14	9	40	ml

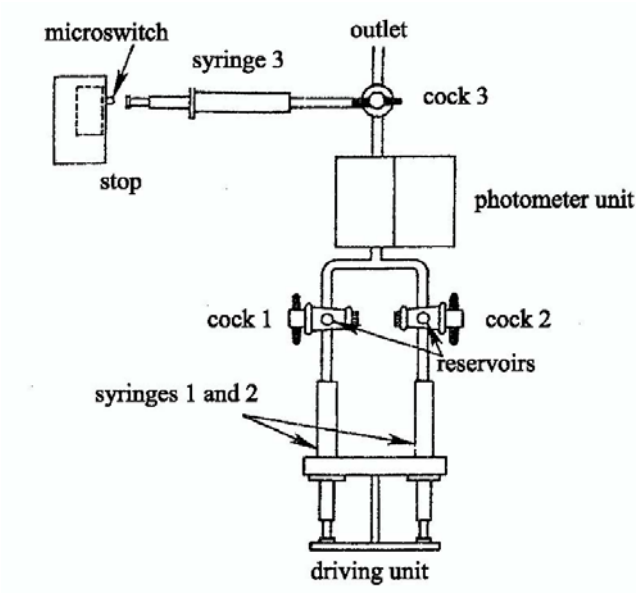

Fig. 1

Sketch of the experimental setup.

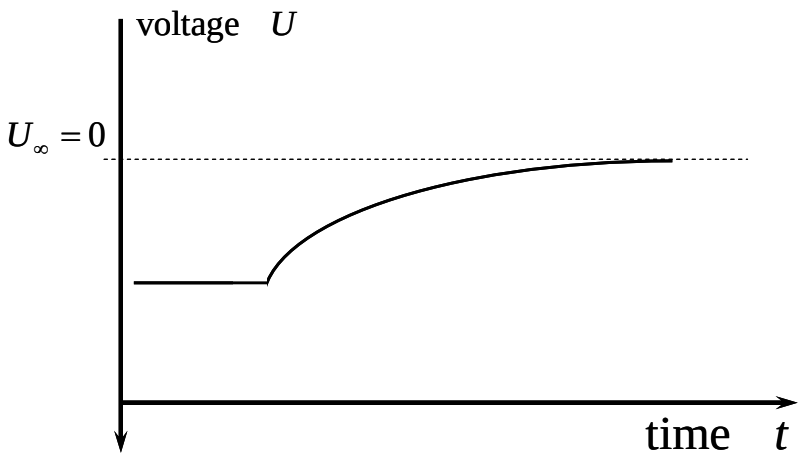

Fig. 2

Sketch of the plot $U(t)$.

Pay attention to the working instructions for handling chemical substance. Pour the waste only in the corresponding, marked bottles.