



## EXPERIMENTAL REPORT

EXPERIMENT N° Test-252

INSTRUMENT D22

DATES OF EXPERIMENT 24. 09. 1997

TITLE **Setup for time resolved neutron small angle scattering (TR-SANS) of liposomes and proteins**

EXPERIMENTAL TEAM (names and affiliation)

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Transient structural changes occur in biological systems in working proteins and at membranes during the development of an electrochemical proton potential (energization), e.g. at liposomes after a pH-jump. In the present work we have developed a novel setup for time resolved neutron scattering (TR-SANS) at the D22 beamline of the ILL.

The active sample, liposomes after a pH jump at pH6.4, was formed by rapid mixing (< 1s) of liposomes (SUV from SBL; 20 g/l, pH8, D<sub>2</sub>O) and acid (1 M MES in D<sub>2</sub>O) using our computer controlled double stopped-flow device (improved version of that in [1] and in preliminary TR-SANS experiments at FZ Juelich (KWS2); used also at ESRF-ID2A and ELETTRA-SAXS for TR-SAXS [2]). For TR-SANS we have chosen a circuit of two stopped-flow drives, a T-valve and an open flow-through quartz cuvette (Hellma, d = 2 mm and 1 mm) shown in fig.1: the cell is filled only to max. 2/3 for the exposure and then evacuated backwards using the T-valve and the stopped-flow drive B, or manually (syringe).

For the coordination of sample process controller (PC +  $\mu$ P or flash) and D22 instrument controller (VME-bus) we have constructed a time-frame controller, which generates also the addresses for the memory pages of the structural film of the working biological system. As a "day-one" solution we used in the experiments Test252 and Test-261 a TTL hardware controller constructed at Mainz. Then this was replaced by an improved programmable ASIC version of the ILL electronics (for experiments 8-03-302 / 1998 and further).

In the Test252 experiment we were successful in the hardware setup and test until a running system according to fig1. After improvements of the data acquisition software first TR-SANS experiments were done 2 month later in the Test-261 experiment (see report and [3])

- 1) Neidhardt, A.; Nawroth, T.; Hütsch, M.; Dose, K. (1991) FEBS Lett. 280, 179-182
- 2) Nawroth, T. et al., (1998) ESRF report LS722 a and b (722\_717) and our WEB-site
- 3) a) Nawroth et al. (1998) DGfB German Biophysics Soc. v.conference.proceedings. 108-

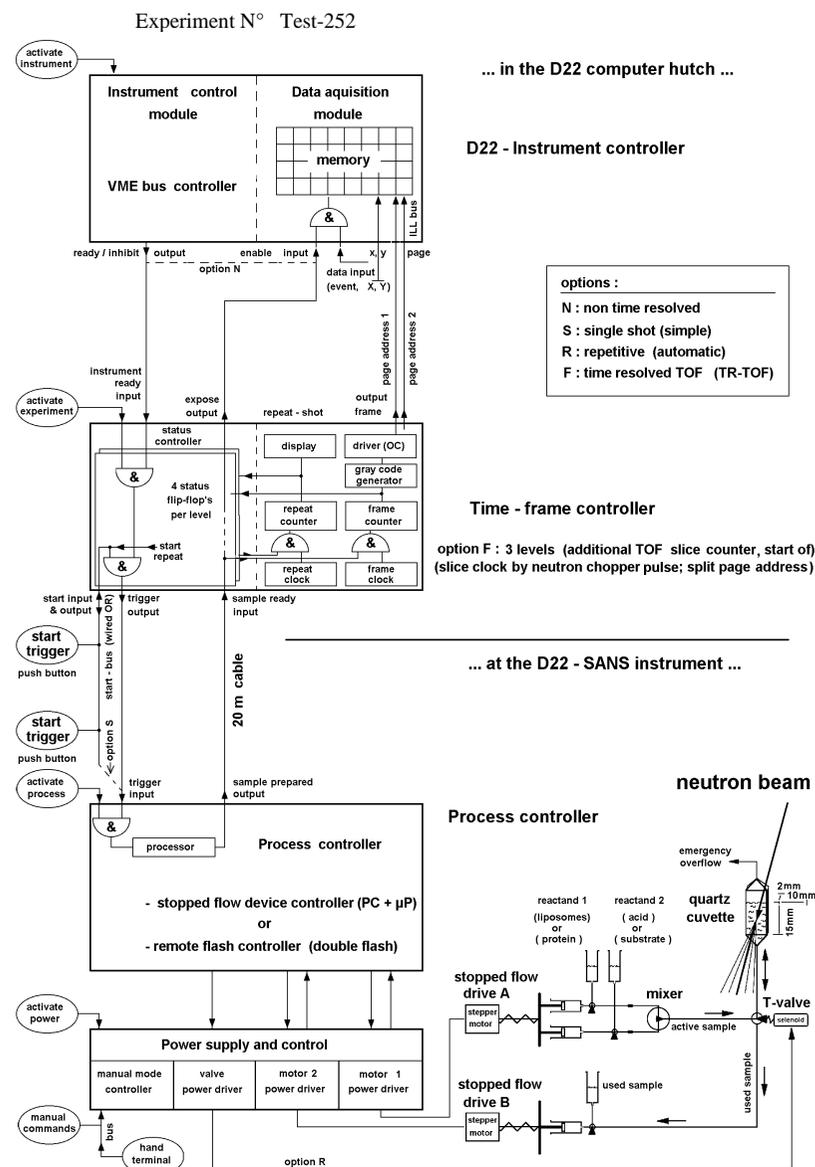


Fig.1: The active sample for time resolved structure investigation by TR-SANS is produced by a computer controlled stopped-flow mixing device or flash. A time-frame controller coordinates instrument and process controllers and generates the memory page