

	<b>Experiment title:</b> Dynamics of critical fluctuations of two liquids	<b>Experiment number:</b> SC685
<b>Beamline:</b> ID10A	<b>Date of experiment:</b> from: 3 May 2000                      to: 8 May 2000	<b>Date of report:</b> 19 September 2000
<b>Shifts:</b> 18	<b>Local contact(s):</b> Federico ZONTONE	<i>Received at ESRF:</i>
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Report:

In this experiment, a mixture of two liquids (nitrobenzene and hexane) was observed close to the critical point corresponding to the unmixing of the two liquid at  $20.29C$ . In coherent scattering condition, the observation of the dynamics of the speckles of the small angle intensity provide direct measurement of the critical-slowng-down observed close to  $T_c$ .

The conditions necessary for carrying out this experiment can be shortly enumerated:  
-High stability of temperature control in order to maintain the sample in the near vicinity of  $T_c$ .

-Measurements at very low  $q$  vectors, ranging from  $.5$  to  $3. \cdot 10^{-3} \text{Å}^{-1}$ .

-Measurements at a high repetition rate in order to be able to observe correlation times from  $10. ms$  to  $.1 ms$ .

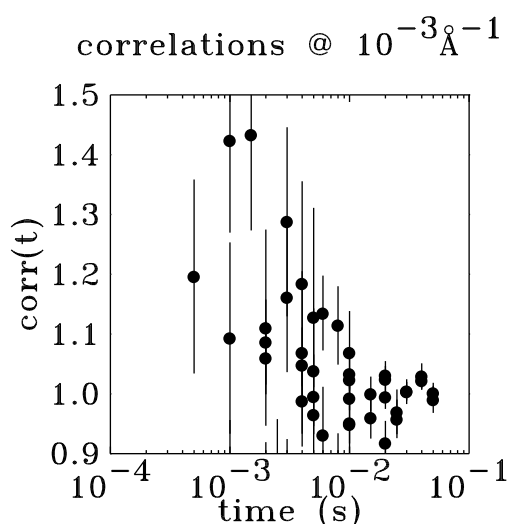
With our setup, the temperature of the liquid sample was stable within  $2.mK$ , and, in practice, measurements were performed at  $T_c+.02K$  to  $T_c+8K$ . Maximum fluctuation length  $\xi$  was about  $1000.\text{Å}$ .

Parasitic scattering from pinhole in the small angle range was difficult to eliminate, and no reliable measurements were obtained under  $10^{-3} \text{Å}^{-1}$ .

Measurements were performed with a beam of high monochromaticity, (Si<sub>111</sub> monochromator) and the intensity was relatively low (close to  $5.10^8$  x-rays/s. The use of "pink beam" in the case of these "very small angle experiments" could improve intensity a factor of one hundred, and this will be soon available at ID10C.

In order to improve statistics, a direct-illumination CCD camera was used, in a photon counting mode (“droplet algorithm”). This provides a very efficient noise suppression in the case of low intensity and large number of images. In order to overcome the (relatively) slow readout rate of our  $1MHz$  converter, the kinetic mode was used. Using slits, only a small region ( $24 * 100$  pixels) was illuminated, and the time correlations were observed on the same frame between regions illuminated at different times during shifting. Tests performed in this mode have shown that a time resolution of  $0.1ms$  was easily obtained. In practice, the shutter was opening with a  $3.7ms$  delay. For this reason, and also because the intensity was small, measurements were carried out only with a  $0.5ms$  minimum time resolution.

The stability of the method was tested, and a statistical data treatment was further carried out, which takes account of: -all experimental rates of measurements, -all expositions on the same frame obtained in the kinetic mode, -all frames measured, -and all the data obtained in the same  $|q|$  domain.



The figure summarizes, for  $|q| = 10^{-3} \text{Å}^{-1}$ , the normalized time correlations observed after statistical treatments. Time is on a log scale. The function has a long time value of 1, and a short time value close to 1.3. Crossover between these two values should occur at about  $1ms$ . Error bars on the figure show that statistics must be improved. All measurements of this figure were carried out over only 2000 frames, and a number of repetitions can be done (each series takes about 5minutes). But major improvement will be obtained from the larger intensities in the “pink beam” setup of ID10C.