

## ZENO: CRITICAL FLUID LIGHT SCATTERING EXPERIMENT

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### ABSTRACT

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The Zeno (Critical Fluid Light Scattering) experiment is the culmination of a long history of critical fluid light scattering in liquid-vapor systems. The major limitation to making accurate measurements closer to the critical point was the density stratification which occurs in these extremely compressible fluids. Zeno was to determine the critical density fluctuation decay rates at a pair of supplementary angles in the temperature range 100 mK to 100  $\mu$ K from  $T_c$  in a sample of xenon accurately loaded to the critical density. This paper gives some highlights from operating the instrument on two flights March, 1994 on STS-62 and February, 1996 on STS-75. More detail of the experiment Science Requirements, the personnel, apparatus, and results are displayed on the Web homepage at <http://www.zeno.umd.edu>.

### INTRODUCTION

At a liquid-vapor critical point the equilibrium density fluctuations which are present in all materials become visible and the clear, dense fluid becomes milky and opalescent in appearance. For some years this has been studied with photon correlation light scattering spectroscopy. The Zeno experiment was to make such measurements in microgravity to overcome the density stratification which limits their accuracy when approaching the critical point closer than 10 mK on earth.<sup>1,2</sup> This report will describe the light scattering layout, and present some highlights from two flights.<sup>3</sup>

### APPARATUS

The availability of an ALV 5000 correlator adapted for our instrument meant that we could make simultaneous measurements at two angles during the flight. It was desirable to make measurements over as broad a range of scattering vectors (angles) as possible, limited only by the necessity of avoiding the forward scattering from the sample cell optics. Thus we came to the two phototube scattering layout shown in Fig. 1. The shutters SH gave a choice of split laser beams to use and thus which phototube saw forward or backward scattered light. Late in the development it was necessary to add filters (F1 and F2) on the shutters to limit the laser intensity into the sample to either 17 mW or 1.7 mW. These powers were chosen to avoid laser induced density gradients at the sample. The scattering angles inside the sample were 10.448  $^\circ$  and 169.564  $^\circ$ . The thermostat TH had three controlled shells around the sample cell. The sample cell was at the center of these shells and had stepped windows whose center sections were adjusted to give a uniform 100 mm section of sample for the laser beam to pass through. This illuminated fluid is the scattering source for the experiment. The inner window surfaces are not coated to prevent reflections so there is a 10% interference modulation of the transmission due to reflections. This provided an essential monitor of the local sample density during the course of the experiment, as is discussed below.

## PERFORMANCE

The Shuttle communications systems provided 83% real-time communication for data downlink and commanding uplink during the flights. The data gaps were routinely played back to us within the next orbit (90 minutes). This level of communication meant that most of the time we operated as if the apparatus was in our lab on earth. Every second we received the next packet of measurements from the instrument and could command and see the measurement response in a few seconds. Thus the instrument computer and telemetry software performed as planned.

One of the most satisfying performance items was the excellent temperature control which we achieved in orbit. With the aluminum experiment enclosure controlled by side and bottom heaters and radiation losses occurring through the top, we routinely achieved  $< \pm 3\mu\text{K}$  (rms, 300 sec averaging) during flight. An example is shown from the first flight in Fig. 2. This four hour period is representative of the time necessary to accumulate 15 to 20 correlograms for a temperature data point.

The principal measurements were of intensity correlation functions. A set of forward scattering correlograms is shown in Fig. 3 covering a span of temperature of 100 mK to 400  $\mu\text{K}$  from  $T_c$  from the second flight.

## RESULTS

Overall we find convincing evidence that we reached the limiting decay rates for  $12^\circ$  and  $168^\circ$  angle scattering as the critical point was approached. In the forward direction the limit found is a factor of two lower than seen in the measurements on earth, as shown in Fig. 4. This reveals the very large errors in decay rates which occur from density stratification on earth. This data together with the back scattering correlograms is being analyzed in terms of the scaled wavevector, the scattering vector times the correlation range, to test the universal predictions for the approach to the limits.<sup>4</sup> These experiments reached scaled wavevectors of almost 100 for the forward scattering with no density stratification across the beam.

The greatest difficulty, and the reason for the second flight, was the extreme sensitivity of the local density to the way the temperature was changed in the experiment: it was possible to generate 1% density errors by moving the sample temperature too quickly while in low gravity. This was not noticed in earlier microgravity studies of critical fluids in the ESA Critical Point Facility. In Zeno these effects were very easy to detect because all the optical signals came from a thin section of sample where the wall induced density changes dominated the behavior. We saw both adiabatic effects<sup>5</sup> and longer term density responses using the cell window interferometer. These effects are largely hidden by surface flows on earth but are easily detected in microgravity. Our strategy for the second flight was to limit these effects by lowering the temperature towards  $T_c$  with ramps whose calculated rate at each stage was low enough not to cause more than 0.1% density change. Following this plan in flight gave excellent stability of the local density as seen by the laser interferometer and scattering signals until we changed the laser path and filter at 1 mK. Then we found the surprise that the 17  $\mu\text{W}$  beam had actually been heating the local region and causing a significant density error. This effect is also well masked on earth.

The excellent temperature resolution was exploited at the end of the second flight in which we recorded the scattering intensity while crossing the phase boundary while scanning at 100  $\mu\text{K/hr}$ . A segment of the forward scattering data is shown in Fig. 5. The least-squares fitting lines for the temperature of the slope break ( $T_0$ ) gave estimated precision of  $\pm 10\mu\text{K}$  ( $\pm 3 \times 10^{-9}$  in reduced temperature). The forward and backward scattered intensity provided two fitted  $T_0$  values which agreed within  $\pm 20\mu\text{K}$ . On earth such a crossing is ten times broader because of sedimentation dynamics giving widths of 200  $\mu\text{K}$ .

The case shown is known to be about 1% below the critical density so this region of the sample is not going through the critical point but is some 1 mK below the critical temperature.

#### ACKNOWLEDGMENTS

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<sup>2</sup>Hannes Güttinger and David S. Cannell, Phys. Rev. **A22**, 285 (1980).

<sup>3</sup>The Zeno flights were: USMP-2 payload on STS-62, March, 1994 and USMP-3 payload on STS-75, February, 1996.

<sup>4</sup>H. C. Burstyn, J. V. Sengers, J. K. Bhattacharjee, and R. A. Ferrell, Phys. Rev. **A28**, 1567 (1983).

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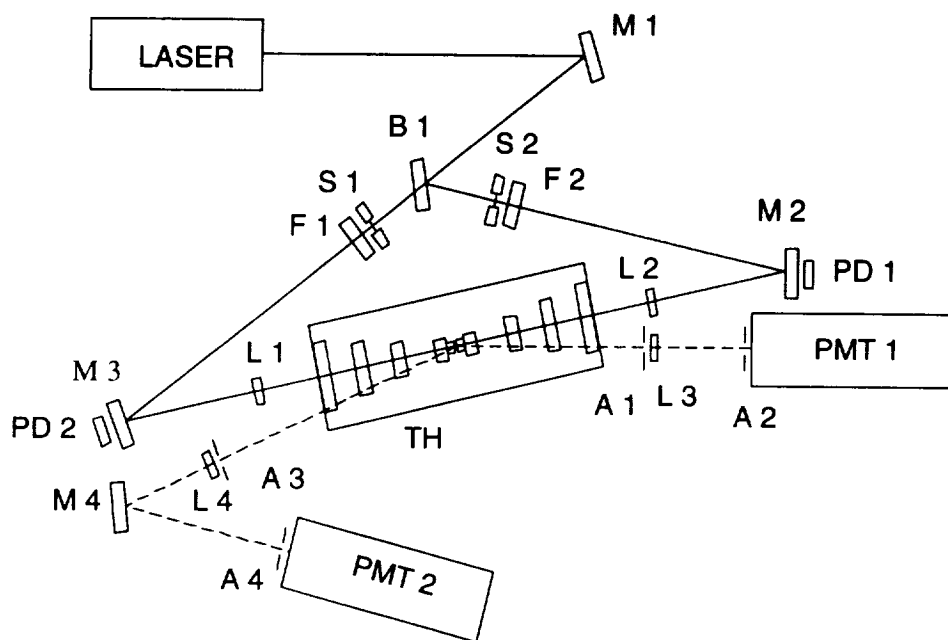


Figure 1. Zeno optical layout. M(i) are mirrors, L(i) are lenses, A(i) are apertures, B1 is a beam splitter, TH is the thermostat containing the sample cell, PMT(i) are photomultipliers, F(i) are filters, and SH(i) are shutters.

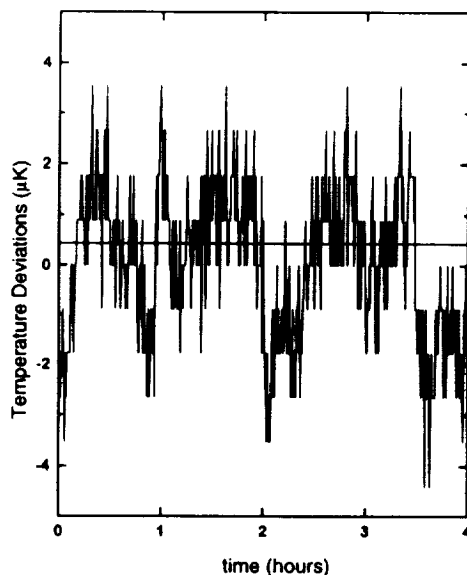


Figure 2. Zeno temperature control during the first flight. Temperature was 1 mK, duration was four hours, sampling averaging was 5 sec. Temperature deviation was 1.5  $\mu$ K rms during this period.

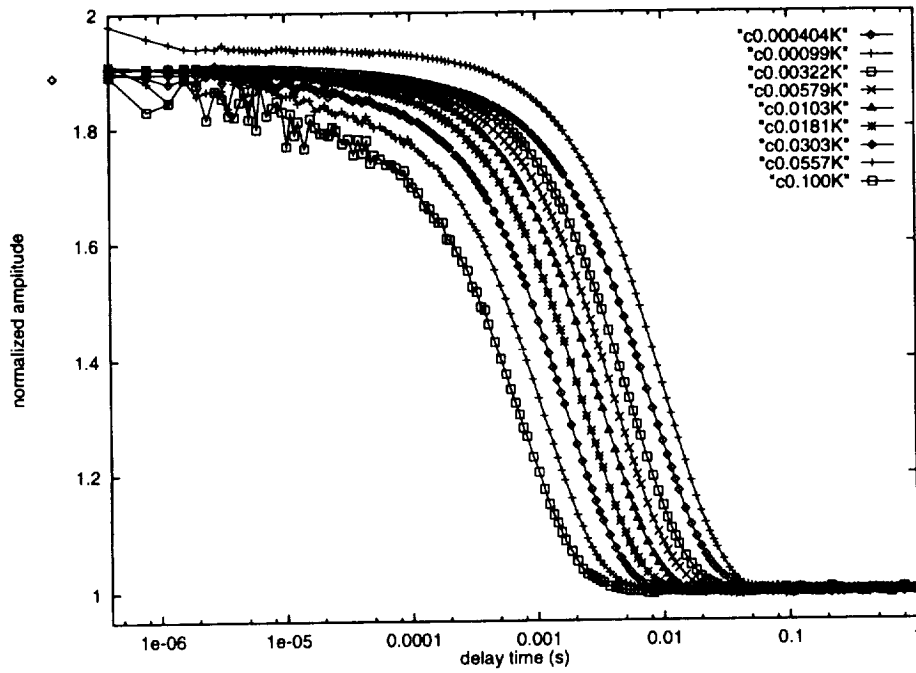


Figure 3. Correlograms for forward scattering,  $12^\circ$ , from Zeno flight 2. For  $(T-T_c) = 100, 56, 30, 18, 10, 5.8, 3.2, 0.99$  and  $0.40$  mK from left to right showing critical slowing down as  $T_c$  is approached.

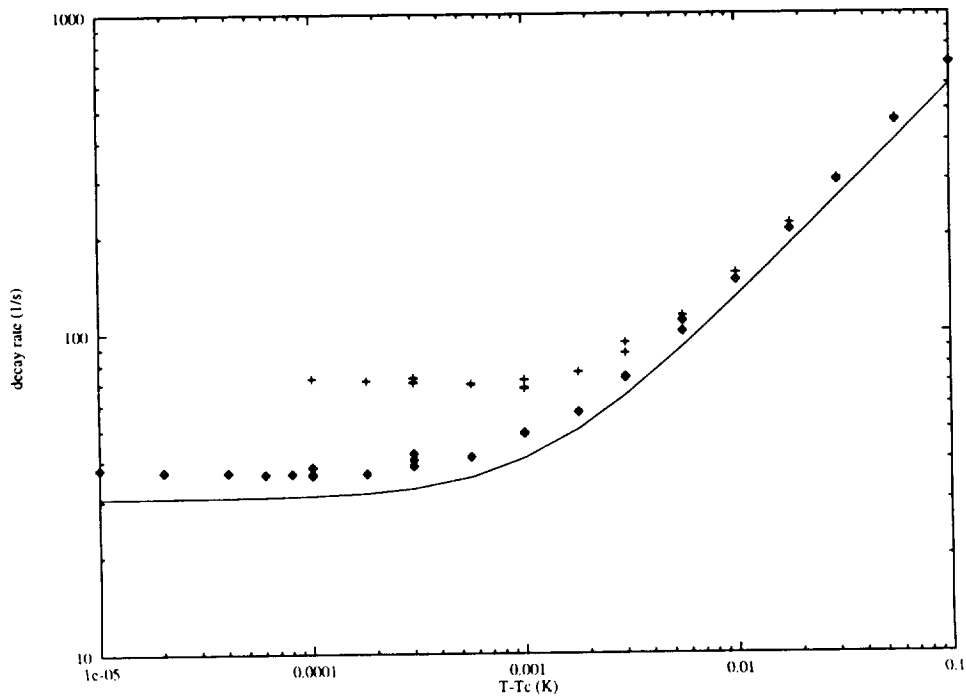


Figure 4. Comparison of ground and flight measured fluctuation decay rates from the first Zeno flight. Curve is an estimated theory line, not fitted. The crosses are ground data and the diamonds are flight data.

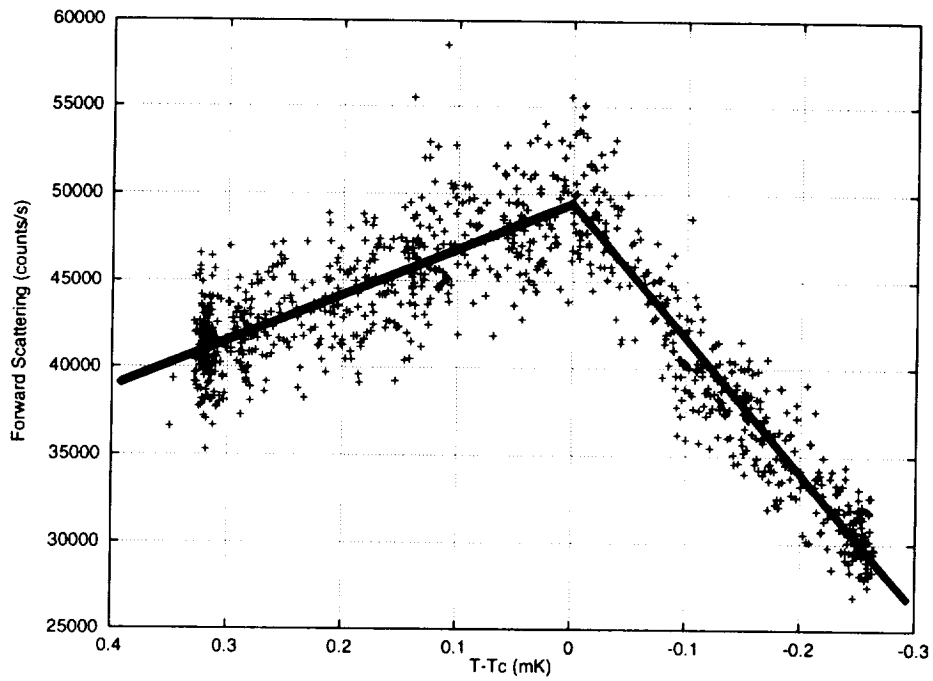


Figure 5. Search for  $T_c$  in micro-gravity. Forward scattering intensity vs. the temperature. The + symbols are unsmoothed, 5 sec. average samples. Scan rate was  $100 \mu\text{K/hr}$ . Plot shows the phase separation by the break in slope of the intensity data.