

# 3 Experimental Equipment

---

Experiments have been performed in a microgravity environment ( $g \approx 0$ ) in space and in the earth's gravity field ( $g = 1$ ) on the ground. The experiments in space were carried out in ESA's Critical Point Facility (CPF) [41], which was flown in the cargo bay of the spaceship Columbia during the SpaceLab IML-2 mission. The experiments in our laboratory at the VWZI were executed utilizing a set up which resembles CPF. The basic concept of CPF originates from our research group at the VWZI, and parts of the flight equipment were actually provided by us. Nevertheless, the instrument as developed by ESA set some limits to the possibilities for our experiment [62]. We start with a description of the facility.

## 3.1 The Critical Point Facility

---

CPF has been developed for the ESA Microgravity Research Program to support scientific investigations into the behaviour of transparent fluids near their critical point. Inputs to its specifications were given by various European and American investigators. It provides to a critical sample the precisely defined and extremely stable thermal environment required to take full advantage of the  $\mu$ -gravity environment, together with a number of optical diagnostic systems. The facility is conceived to run experiments automatically from a predefined timeline, but it supports a space to ground communication system allowing to monitor the experiment in real time and to modify this timeline from earth.

### 3.1.1 Outline of the CPF

---

The CPF consists of two interconnected units: the experiment drawer and the electronic drawer. Core of the experiment drawer is the thermostat chamber, into which exchangeable Thermostat Units (THU's) can be mounted. Each THU holds an experiment-dedicated Sample Cell Unit (SCU). During IML-2, CPF was located in the upper part of a 19 inch SpaceLab Rack when five

THU's were used for five different experiments. The exchange of THU's between experiments was performed manually by a SpaceLab crew member.

The THU chamber is located between the Optical Input and Output Systems (OIO). The experiment drawer furthermore houses the Analog Electronic Box, which contains the THU Thermal Control System (TCS) and electronic drivers for other SCU directed operations (Experiment Dedicated Equipment: EDE). The electronic drawer contains highly stabilized power converters and the central Data Handling System (DHS).

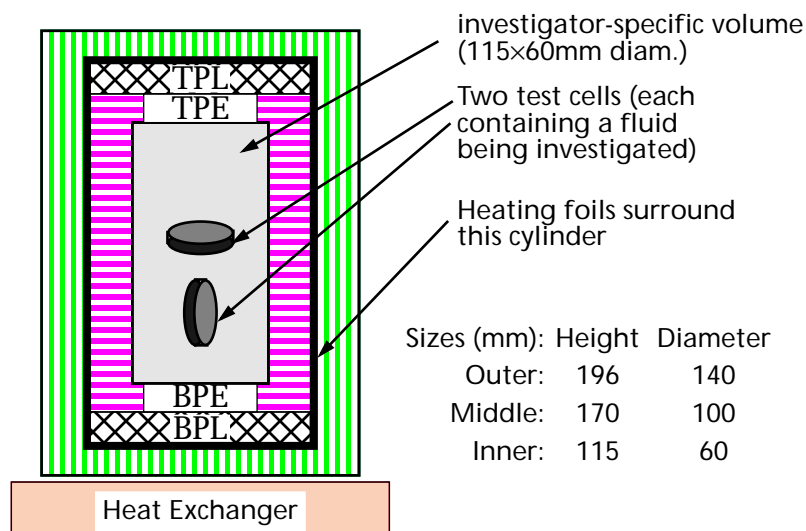
The CPF runs experiments according to a predefined programmed timeline that has been defined by the corresponding investigator and loaded into the CPF's memory. This timeline can be modified in real-time from the ground during the experiment's run, when requested by the investigator.

The various elements mentioned in this outline are described in the following sections.

### 3.1.2 The Thermostat Unit

The THU (fig. 3.1) is cylindrically shaped (height 196 mm; diameter 140 mm) and uses the “gradient reduction” principle [63]. This implies that only one main temperature sensor is employed for regulation purposes; temperature gradients are reduced by proper mechanical construction, proper arrangement of heaters and the use of thermocouples and Peltier elements as differential sensors. Low thermal resistivity of the structure and the use of Peltier elements, both between THU and the investigator-specific volume and between THU and a heat exchanger, enable fast changes of the temperature set point ( $T_{set}$ ) of the thermostat and thus substantially reduce the experiment duration. In addition to providing thermal control, each THU has optical and electrical interfaces to enable stimuli and diagnostics to interact with the test fluid.

Figure 3.1 A simplified cross-section of the THU and HEX.



The actual THU consists of three coaxial aluminium shields, the middle one being surrounded by Joule heating foils, with torus-shaped Peltier elements on the top (TPE) and bottom (BPE). The two inner shields are mounted directly onto one baseplate (BPL). Between the Peltier ele-

ments on baseplate and topplate (TPL) a cylindrical SCU can be mounted, with a length of 115 mm and a maximum diameter of 60 mm. To increase speed of exchange of heat with the environment, when mounted in its chamber the THU is pressed against a Peltier driven heat exchanger (HEX). The HEX controls the main heat input/output from ambient to the THU. For temperature regulation and operation of the EDE, the THU is equipped with three p24 electrical connectors which are engaged automatically when the THU is inserted in CPF.

### 3.1.3 A Sample Cell Unit

---

When using the CPF the investigator has to deliver his sample in a (dedicated) SCU. If use is made of the interferometer diagnostics, the facility designed Interferometer Unit (IFU) forms part of this SCU; the remaining part, hereafter referred to as Sample Cell (SC), may be defined by the investigator, within the specifications of the interface control document [62].

For temperature stabilization and control the SCU is mounted in the investigator specific volume of the THU (see fig. 3.1). For IML-2 the CPF THU design is identical for all experiments; thus all THU's have basically the same thermal regulation characteristics. Small differences in SCU design are accounted for by the capability to adapt certain regulation parameters after mounting the SCU into the THU.

For operation of the TCS, the SCU should contain three temperature sensors but no heaters; the sensors are required for regulation (SCUr), monitoring (SCUm) and overheating protection purposes. It is, however, possible to integrate sample heaters, additional sensors and/or other EDE in the SCU<sup>†</sup>. The regulation sensors and the EDE harness between SCU and THU are part of the SCU. Two SCU's have been designed for the experiments described in this thesis, which are discussed in section 3.3.

### 3.1.4 The optical diagnostics

---

The optical diagnostic systems present visual data by means of which the thermodynamic phenomena in the samples can be explored. There are two sources of illumination, green (555 nm) light-emitting diodes (LED's) and a 1 mW laser beam at 632.8 nm. The laser beam is split into a wide beam (12 mm dia.) of 0.06 mW and a narrow beam (0.6 mm dia.). These light sources are combined within the optical system to form two observation channels. Their geometry with respect to the THU is shown in fig 3.2. Of the optical components in this figure that are merely numbered a list is given in table 3.1.

In the upper channel, interferometry (IF) images (Twyman-Green type [64]) are formed using the wide laser beam. The input of the laser beam is on the side of the THU and the output of this IF channel is on top. The IF images are recorded by means of a CCD camera and a Minolta 9000 still camera. The field of view is circular with a 12 mm diameter. In the present configuration, the IF system is not optimized for our SCU. The implications of this are discussed in chapter 4.

---

<sup>†</sup> The use of such EDE may however, in some configurations, slightly degrade the quality of regulation.



**Table 3.1** List of optical components.

<b>nr.</b>	<b>component</b>	<b>nr.</b>	<b>component</b>
1	Polarizing beamsplitter cube	13	Selfoc fibre guide
2	Mirror	14	Rotating disk/ selector disk
3	Shutter	15	Objective
4	Beam expander	16	Absorber
5	Window	17	Neutral density filter
6	Retardation plate	18	Diffusor
7	Diaphragm	19	Baffle
8	Beamsplitter	20	Green filters
9	Reference fibre + diffusor	21	Spike filter + beam stop + baffle
10	Bandpass filter	22	Pinhole - shutter mechanism
11	Lens	23	Beam monitor diode
12	Optical connector	24	

In the lower channel, several types of measurement are conceivable. At the input of this channel, on the side below the IF channel input, one finds both the LED's and the narrow laser beam. The LED's allow direct visual observation of the sample by the same cameras that are utilized to record the IF images. The field of view is circular with a 12 mm diameter. The phenomena of critical opalescence and bubble formation in the two-phase region may be observed this way. The video (CCD) camera and/or a photcamera acquire the images from either the direct visualisation or the IF channel as selected by the automatic timeline program running the CPF.

The narrow laser beam is utilized to study light scattering by the sample. Small Angle Light Scattering (SALS) data from  $0^\circ$  to  $30^\circ$  is collected with a dynamic range of  $10^4$  by means of a linear diode camera (LDC). The 20 first pixels of this LDC measure the beam attenuation in transmission (turbidity). In the SALS diagnostics, the investigator selects at any time the number of scans that the LDC will make, with an average calculated based on 1 to 256 scans. The investigator also selects the scan frequency, from 13.3 to 1.66 kHz. Unfortunately, the SALS system including the turbidity did not function properly during the IML2 mission and no worthy results could be elicited from the SALS data.

Wide Angle Light Scattering (WALS) is collected at 7 discrete angles between  $-38^\circ$  and  $90^\circ$  by means of a photomultiplier tube (PMT). The WALS set up consists of eight optical fibres, which are periodically scanned at 0.1 Hz by the PMT using a selector disk; seven fibres at the discrete angles and, for laser intensity calibration, one fibre to monitor the intensity of the input laserbeam with an accuracy of 0.05%. Besides these scans every 10 seconds the dark current is measured. The WALS measurement is the integration of the photomultiplier signal over the time that it is exposed to a fibre output, i.e. 250 ms.

The performance of these methods is listed in Table E.2 in Appendix E.

### 3.1.5 The Thermal Control System

Thermal control is based on thermistor temperature sensors (YSI precision) mounted in a wheatstone bridge. A thermistor is a semiconductor device, the electrical resistance of which varies strongly with temperature; nominally, at 45°C the resistance is 4600 Ω, with a temperature coefficient of approximately 4%/°C. The bridge consists of 5 ppm/K resistors (Philips MPR24) and is stabilized further by locating it in a recess in the THU just below the BPL. Two thermistors, one in the BPL and one in the SCU (SCUr), are involved in the regulation circuitry as main sensor for two different modes of operation; a third one, located in the SCU (SCUm), is not part of the regulation circuit but act as an independent monitor of the SCU temperature.

The secondary sensor system consists of three differential thermocouples and two Peltier elements. The Peltier elements actually consist of a large number of thermocouples in series, yielding a sensitivity at least equal to that of the thermistor bridge. Their advantage over the latter is that they are “passive” sensors, hence they do not generate heat. However, they can only be used to measure a temperature difference between their two end plates. The thermocouples monitor the temperature differences between the BPL and TPL and the BPL and upper and lower halves of the 2<sup>nd</sup> shield (the Outer Thermal Shield: OTS) respectively; the Peltier elements (TPE & BPE) in fact measure axial temperature gradients across the SCU.

A complex algorithm converts the output of the sensor circuits to inputs for the PWM (Pulsewidth Modulated) input currents for the heaters on the OTS and for the TPE & BPE. Pulsewidth modulation is required to minimize the power budget for the system; however, to reduce EMI inside the THU the heater current is routed via a PWM → DC circuitry.

The electric circuitry and the microprocessor and software for these operations are located on five PCB's in the Analog Electronics Box.

There are two modes of operation:

**MODE 1: *Coarse Mode***, used for quick heating up or cooling down. The BPL thermistor is the main sensor, the TPE & BPE are used to monitor the temperature differences between SCU and TPL and BPL and to enhance the reduction of these differences.

**MODE 2: *Fine Mode***, used in stable operation for accurate control of the SCU temperature and the gradients across it. The SCUr is the main sensor, the TPE & BPE are only used in the sensor mode.

In the fine mode the SCU temperature can further be changed in a controlled way according to two scenarios: a quench or a ramp. A **quench** is a fast, precisely defined temperature step up or down. It is executed by changing the  $T_{set}$  and in parallel activating the TPE & BPE for the time required to move the corresponding amount of heat to or from the SCU. In a **ramp** the  $T_{set}$  is 'continuously' changed by small steps so as to make the SCU temperature change linearly in time.

With this arrangement the following performances are obtained.

- Range of operation: 30 → 70 °C
- Mode 1 heating/cooling rate 36/10 K/hr
- Power dissipation @ 45 °C: (typ.) 7 W
- Mode 2 stability @ 45 °C (static or in ramps): < 20 μK/hr
- Mode 2 temperature gradient (at the sample): < 10 μK/cm

- Quench steps: 0.1 mK below 1 mK,  
1 mK for 1-100 mK
- Quench rate: 25 mK/s
- Minimum speed of ramps: 0.3 mK/min.

### **3.1.6 The Experiment Dedicated Equipment**

---

The CPF contains a number of devices, which are not considered to be part of the basic set of stimuli or diagnostics of the facility, but are implemented on request of one or more users. This Experiment Dedicated Equipment (EDE) is part of the CPF design, but the actual device is provided by the user; it is only accessible through an electrical interface. Of these EDE the following elements are - for functional reasons - more or less integrated in the TCS:

- three additional thermistors;
- a Voltage Source System (VSS);
- a Current Source System (CSS).

The bridges for the additional thermistors are designed to match the same type of thermistor as used in the TCS. The three thermistors can individually be switched to “high sensitivity” mode (power dissipation = 12  $\mu$ W), “low sensitivity” mode (power dissipation = 0.75  $\mu$ W) or off. Additionally, one thermistor can be switched to “pulse heating”; in this mode the sensor is used as a heater, delivering 5.65 mW of power to the sample. The thermistors – as EDE – are operated from the timeline. All three thermistors have been utilized in our set up.

The VSS is a system, that is intended to deliver to the SCU a calibrated voltage of up to 500 VDC, e.g. for creating an electrical field in the sample fluid; it is not allowed to draw any power from it. The VSS has not been utilized in our experiment.

The CSS is intended to supply a well defined amount of heating power to the sample, by delivering a calibrated electrical current to a resistance heater inside the cell. This current ranges from 0 to 1 Amp. in steps of 20  $\mu$ Amp. The CSS has been utilized in our experiment.

### **3.1.7 The Data Handling System**

---

During the time that CPF experiments are running on board Spacelab, the investigator receives telemetry data on the ground to monitor the progress of the experiment and to provide a basis for real time decisions concerning the subsequent execution of the experiment.

All the scientific and housekeeping data generated by the CPF are displayed in real time (or replayed after every period of loss of contact with Spacelab) and updated once per second. The video images from the CPF are displayed in real-time, at the television rate (30 frames per second) during short, pre-selected periods, and continuously at a reduced rate of one image every six seconds. Voice contact with the crew can be established while the crew is executing CPF-related tasks. All data sent to the ground is recorded and made available to the investigator after the mission, together with the pictures taken by the camera. This may take some time though, usually about half a year.

Although the CPF runs automatically according to a predefined program, the investigator can also interact with the CPF during an experiment on the ground or during the mission, to modify

the way that the experiment is running and in particular to modify the predefined program. While carrying out the experiment, the onboard operation itself can be modified by issuing commands from a dedicated microcomputer, part of the CPF command Electrical Ground Support Equipment (EGSE). This feature of the CPF has proven to be absolutely essential for reaching the scientific objectives of our experiment.

Additional to the NASA site in Huntsville (Al, USA), from which the modifications to the timeline were executed, the course of our experiment was monitored by a site in Amsterdam, called DUC (Dutch Uttilization Center). As part of the remote centers project by ESA, under project-name CRESCENDO (Center for REmote Science ENhancement by DUC Operations), the National Aerospace Laboratory (NLR) established a site at NLR Amsterdam to enable quick, preliminary analysis of the scientific and housekeeping data. In fact, the real time decisions to modify the experiment timeline were based mainly on the result of this analysis.

### 3.1.8 The automatic timeline program

All the timelines are predefined by the investigators and are stored in the CPF's memory before the launch. As soon as the crew has inserted a THU in the CPF and initiated the corresponding experiment, the CPF runs automatically according to that timeline. This automatic timeline program is called the Experiment Parameter Table (EPT). Each experiment has an EPT. Each EPT consists of a header and up to 1024 sequential steps called action points. The header specifies information such as the value of  $T_c$  and the duration of the experiment, along with parameters for regulation and quench purposes. Each action point defines the time that the action will last and up to four commands that control the stimuli/diagnostics of which the first is reserved for temperature control purposes.

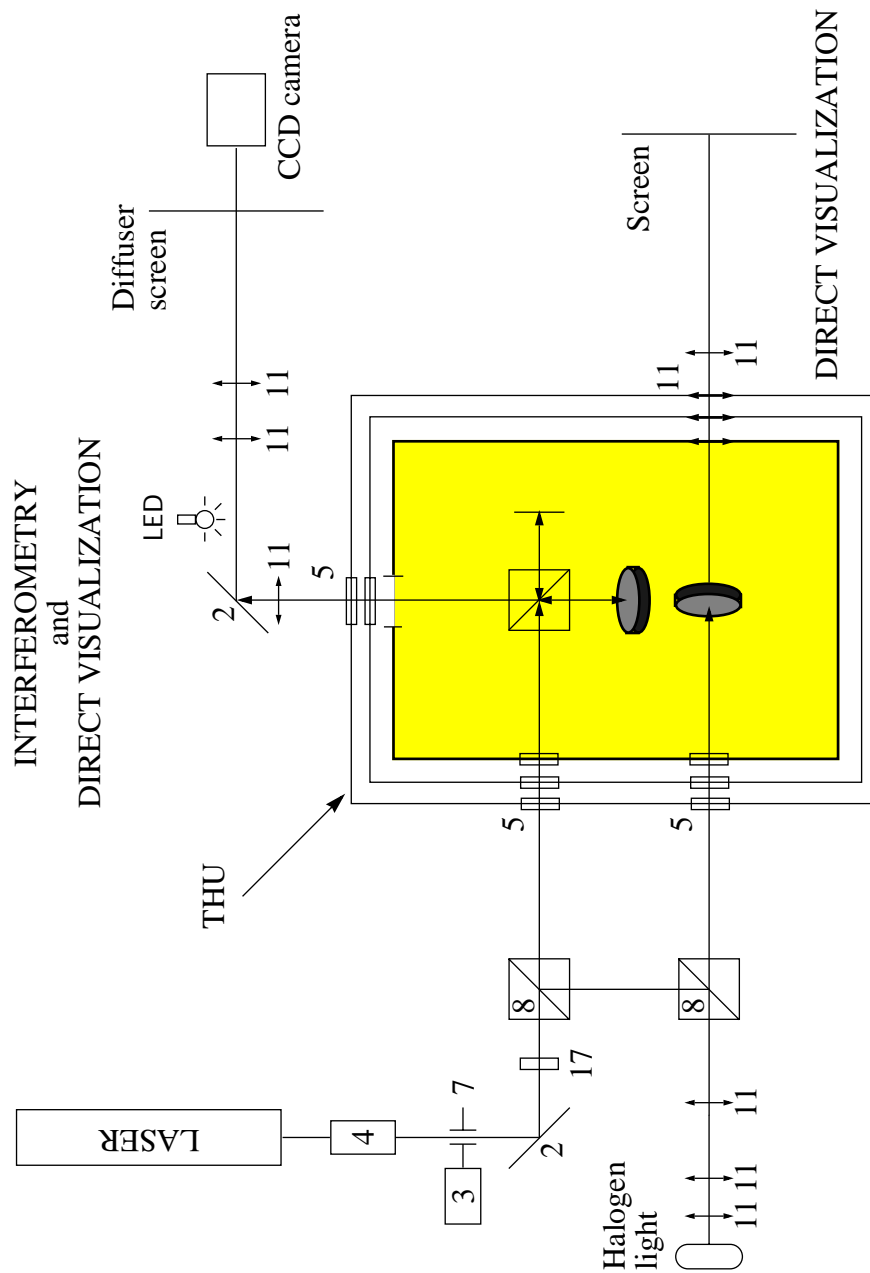
## 3.2 The Laboratory Equipment

The set up at the VWZI served several purposes; to prepare the MIM3-SCU for experiment, i.e. fill it at critical density, and to perform two experiments, being the one to study the transfer of heat at  $g=1$  and the one to investigate the relation between the density and the refractive index. The light scattering experiments are not suited for a “on ground” comparison because of the influence of the gravitational density gradient (eq. (2.67)) near CP on light scattering [65-69].

As mentioned in the introduction to this chapter, the lab equipment resembles the CPF. The optical arrangement in relation to the THU is displayed in fig. 3.3, of which the numbered components correspond to those listed in table 3.1. The optical diagnostic methods of the lab equipment are direct visualization by illumination of the sample with halogen light and interferometry of the Twyman-Green type where use is made of an expanded He-Ne laser beam (wavelength of 632.8 nm). The image that originates from illumination by halogen light is magnified largely on a screen. The IF images are recorded on video along with the time in tenth of seconds. In order to correlate the video images to the temperature data provided by the TCS, a LED is placed in the output path of the IF channel that is flashed each time a heat pulse is initiated.



Figure 3.3 THU and ground optical arrangement.



The ground-TCS is identical to the CPF-TCS. Consequently, the ground-TCS is able to regulate a THU (including SCU) that is designed for CPF and offers the same thermal capabilities and EDE stated in section 3.1. In contrast to the CPF, the ground facility does not run according to a predefined timeline; each action, such as a temperature change or a heat pulse, needs to be communicated to the TCS manually through a computer. A program called ‘Snoopy’ issues these commands to the TCS and stores the temperature and housekeeping data at a rate of once per second.

The filling of the SC’s was performed with a standard filling set up. The filling equipment was connected to a SC either by a valve or a capillary, depending on the SC concerned (see the description of the SC’s in the next section). The SC’s were filled inside the THU in order to control the temperature accurately. The connection between SC and filling equipment was possible through a small opening in the baseplate of the THU.

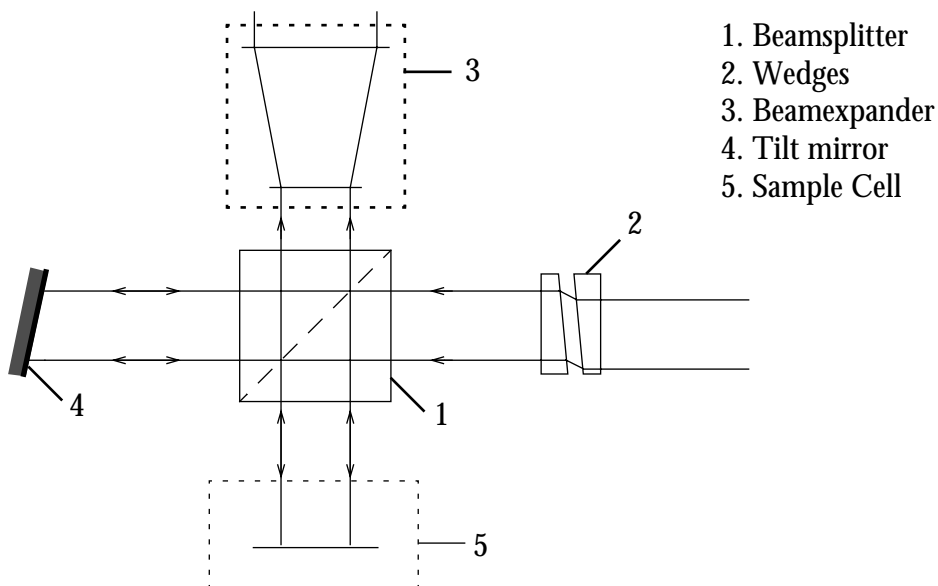
### 3.3 The Sample Cell Units

Two SCU's have been designed for the experiments described in this thesis; the SCU that was utilized for the space experiment, referred to as MIM3-SCU after ESA's namegiving to the experiments on CPF, and the SCU for the investigation into the relation between the density and the refractive index of  $\text{SF}_6$  near its CP, hereafter referred to as DER-SCU. Thermistors enable the measurement of the temperature with a sensitivity of  $10 \mu\text{K}$ . The MIM3-SCU accommodates of the EDE three additional thermistors and the arrangement for the CSS. In the following sections the several parts of these two SCU's are presented.

#### 3.3.1 The interferometer unit

When use is made of the interferometer diagnostics, one finds the IFU on top of the SC. In the centre of this unit, a cube beamsplitter divides the incoming laserbeam into parts of equal intensity, downwards to the SC and sideways to a mirror. The reflections are recombined by the beamsplitter resulting into interference. The quality of the interferogram is determined by the surface quality of all optical elements (including those in the SC); their quality is pitch polished, 20-10 scratch-dig [70], and their surface flatness is smaller than one tenth of the wavelength of the laser light. Figure 3.4 shows a schematic drawing of the optical set up for the IFU that was designed for our SCU's.

Figure 3.4 The optical set up for the IFU.



1. Beamsplitter
2. Wedges
3. Beamexpander
4. Tilt mirror
5. Sample Cell

The elements in this drawing specific to the IFU, i.e. 1 to 4, are regarded below. The two sample cells (5 in fig. 3.4) are discussed in sections 3.3.2 and 3.3.3.

The cube beamsplitter (1 in fig. 3.4) consists of matched pairs of right angle prisms cemented together. The hypotenuse of one prism has a 50% reflection coating. According to the specifications [70], the beam deviation is within 3 arc minutes. The part of the laserbeam that is directed towards the sample cell is required to be parallel to the optical axis within 1 arc minute (as will be discussed in section 3.3.2) so that a correction of the beam direction is needed. The correction of

the beam direction is cared for by two wedge prisms (2 in fig. 3.4) at the laser light-entrance of the IFU, in front of the beamsplitter. Pure geometrical optics implies that by combining two equal wedges in near contact, and independently rotating them about an axis parallel to the normals of their adjacent faces, a ray can be steered in any direction within a narrow cone determined by their wedge angle.

Primarily, the field of view is determined by the smallest opening in the path of the laserbeam which is 12 mm. When the resulting interferogram is projected at maximum size on a film of 500×500 pixels, a resolution of at best 24 μm/pixel is reached. To improve the resolution, a beamexpander (3 in fig. 3.4) that expands a factor 3 is utilized, resulting in a field of view of 4 mm. The tilt mirror (4 in fig. 3.4) in the IFU is adjustable in order to obtain the desired number of fringes and the angle of them with respect to an arbitrary axis in the interferogram.

### 3.3.2 The Spaceflight Sample Cell Unit

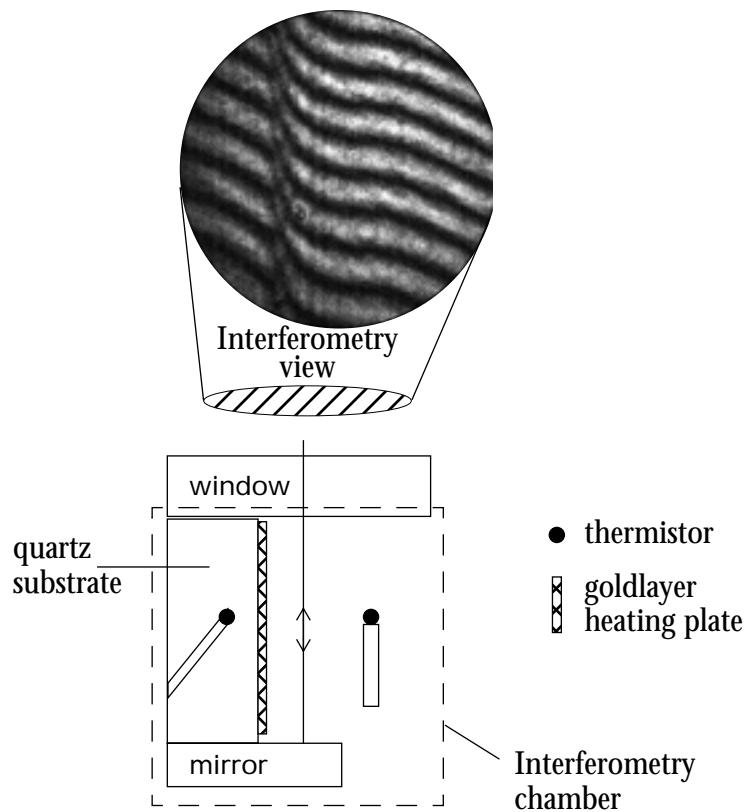
---

Our spaceflight SCU is designed for two different types of experiments and, therefore, it houses two chambers for the experiments that are performed. The experiment that looks at the transfer of heat by imposing a plane thermal disturbance to the fluid is performed in what we refer to as the interferometry chamber. The experiment in which the scattered light from a laserbeam is collected is performed in the scattering chamber.

**THE INTERFEROMETRY CHAMBER.** The core of the interferometry chamber is the heating plate. It is a very thin layer of gold deposited on a quartz substrate. The heat is generated by running an electric current through this layer utilizing the CSS. Density changes in the fluid adjacent to the gold layer are monitored using the IF images. As a part of the interferometry system, this chamber accommodates a mirror. One part of the laser beam that builds the interferogram enters the fluid parallel to the gold layer and is reflected by this mirror so that it passes along the heater a second time and leaves the fluid through the entrance window. Inside the interferometry chamber, the temperature is measured by two YSI precision thermistors as EDE; one in the fluid and one behind the heater in a mounting hole inside the quartz substrate. The third EDE thermistor is placed in the housing of the IFU to measure the SCU's temperature at an additional location besides the locations of the SCU<sub>r</sub> and SCU<sub>m</sub> (see page 32). A schematic of the interferometry chamber is displayed in fig. 3.5.

The heater substrate is cut out of a synthetic quartz cylinder of 20 mm diameter and 16 mm height at 1 mm distance from and parallel to the symmetry axis. The field of view of the interferometer allows observation of the fluid up to a distance of 3 mm normal to the heater. The gold layer is deposited by vaporization and measures a height of 14 mm, is 18 mm wide and 20 nm thick, except for two 1.5 mm wide and 1 μm thick edge layers. Wires are connected to the heater with silver epoxy through the edge layers. The total resistance of the gold layer  $R$  is measured to be 3.6 Ω. To the bottom of the heater substrate a 4 mm thick piece of synthetic quartz is optical contacted, coated with silver by vaporization to serve as the mirror. The mirror surface flatness is smaller than one tenth of a wavelength and is perpendicular to the heater within 1 arc minute. This quality is necessary since we want a ray of laser light to cover a fluid layer smaller than the resolution of the IF images which is of the order of 10 μm. For a 16 mm path travelled twice, this leads to the requirement for the heater to be parallel to the optical axis within 1 arc minute and for the mirror to be perpendicular to it within the same accuracy. The window is 8 mm thick and has a surface flatness at both sides smaller than one tenth of a wavelength.

Figure 3.5 Schematic of the interferometry chamber.

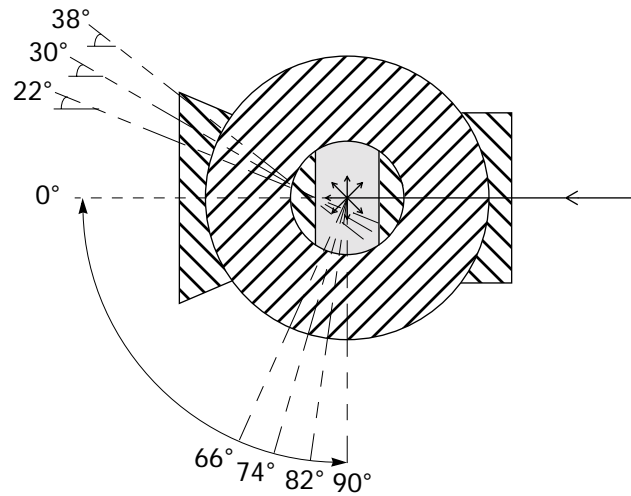


The thermistor in the fluid is located approximately 9 mm from the heater. The time constant (the time required for a thermistor to indicate 63% of a newly impressed temperature) is 1 second in well stirred oil and 10 seconds in still air. In  $\text{SF}_6$  of critical density the time constant has been measured to be about 3 seconds. In appendix D the response of the thermistor to changes in the bulk temperature of the fluid is derived. The outcome is that the surface temperature of the thermistor should follow that of the bulk fluid more and more closely as  $T \rightarrow T_c$ . The thermistor in the substrate is located 4 mm from the heater. Unfortunately, due to an extreme offset of this particular thermistor, it could not be used at its highest sensitivity. For the impact of this see section 6.2.2.

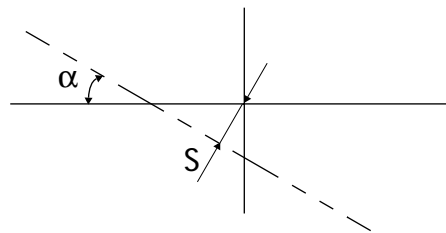
**THE SCATTERING CHAMBER.** The scattering chamber enables light scattering measurements as well as direct visualization (see fig. 3.2). A top view of the scattering chamber is displayed in fig. 3.6. The arrangement of the WALs optical fibres is indicated as well, in contrast to the SALS arrangement which did not function.

The scattering chamber is manufactured completely from synthetic quartz (Homosil). It is a hollow cylinder with inner diameter of 10 mm, outer diameter of 25 mm and of 15 mm height. Both on the inside and outside, additional segments are glued in order to eliminate refraction of the main beam at the cylinder walls. With these segments the width of the chamber becomes 5.6 mm. The WALs fibres for the large angles ( $66^\circ - 90^\circ$ ) are directed towards the centre of the chamber. In positioning the WALs fibres for the light scattered over smaller angles ( $22^\circ - 38^\circ$ ) it is accounted for the refraction at the flat interfaces. The direction of these fibres is sketched underneath the top view in fig. 3.6.

**Figure 3.6** Top view of the scattering chamber including the arrangement of the WALS optical fibres.

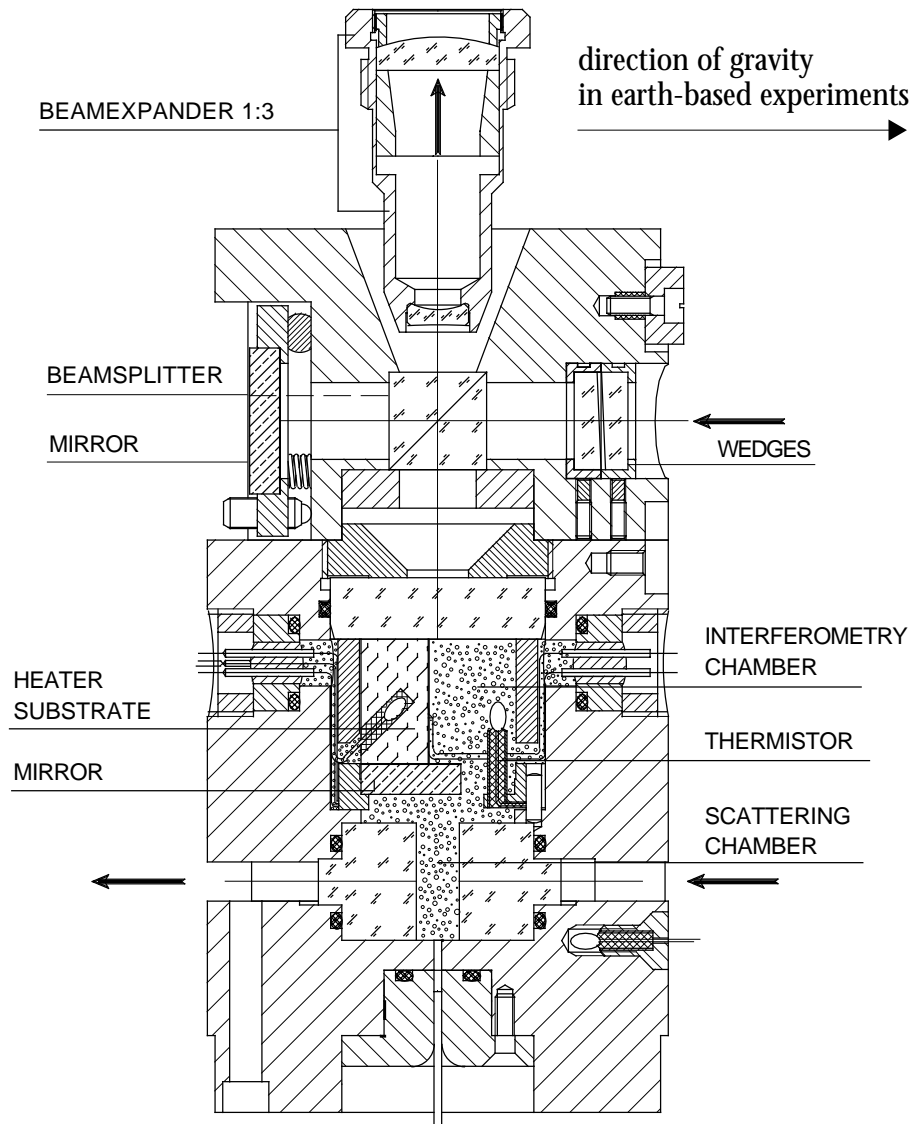


$\alpha$	S(mm)
22°	1.54
30°	2.20
38°	2.98



In the spaceflight SC, the two chambers are interconnected amounting to a total volume of approximately 6 cm<sup>3</sup>. The complete spaceflight SCU is displayed in fig. 3.7. Two basic requirements precede the design towards an experiment dedicated SCU: the requirement for the SC to withstand the pressures that may be expected for a fluid near to its critical point and the requirement to be able to fill the SC to the critical density. For the SC material aluminium was chosen because it combines a high thermal conductivity with a relatively low heat capacity accelerating the equilibration of the SC after a temperature change of the THU. Furthermore, it has a relatively low specific weight and is easy to manipulate. With regard to the second requirement, in section 2.4.3 it is explained that the position of the meniscus is a measure for the distance to the critical point. When the meniscus disappears or reappears at the volumetric middle of the SC on crossing  $T_c$  respectively from below or above, the density of the fluid sample is critical. Therefore, the SC is designed to be symmetrical about a plane perpendicular to the field of view.

Figure 3.7 The Spaceflight SCU.



Altogether six wires are present inside the SC; two for each of the two thermistors and two current leads to the goldlayer. These wires are led through the walls of the SC by a four pin and a two pin lead through respectively. These lead throughs are meant for vacuum purposes but are pressure-tested up to 100 bar. Instead of a valve it is chosen to use a capillary to fill the SC. Up to the moment of the construction of this SC, there was no valve available that was leak-tight over a period of at least a year.<sup>†</sup> All other openings are closed by viton O-rings.

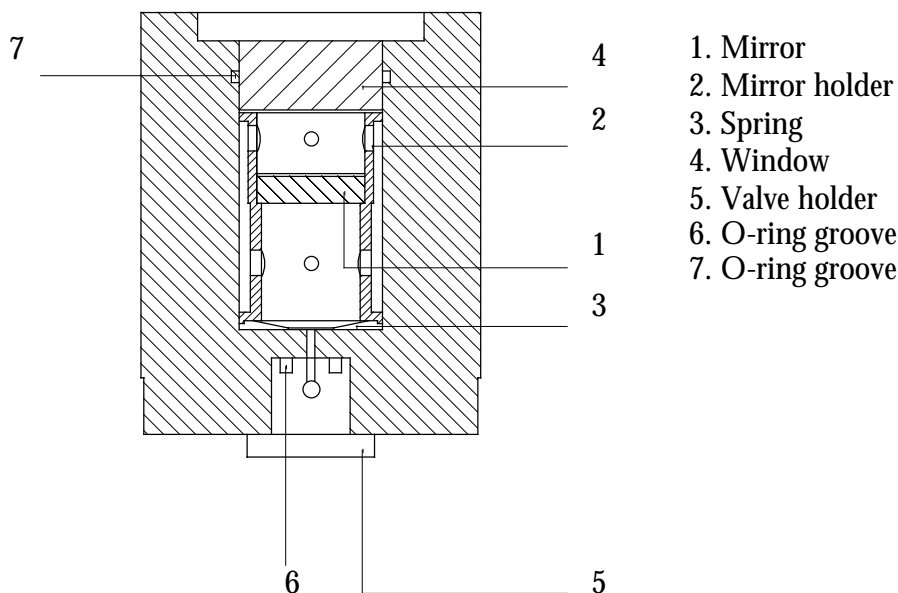
### 3.3.3 The Sample Cell Unit for refractive index measurements

The experiment in which the relation between the density and the refractive index is determined is carried out with the DER-SCU. Here, too, interferometry is used and thus an IFU is part of the DER. The same IFU as in the MIM3 is utilized, but without the wedges and the beamexpander.

<sup>†</sup> For logistic reasons, NASA requires the SCU to be mounted in the spaceshuttle half a year prior to the launch.

The aluminium SC is designed to allow easy calculation of its interior volume and to be symmetrical about a plane perpendicular to the field of view. It houses one large chamber that accommodates the obligatory mirror, as displayed in fig. 3.8. This mirror is glued to a mirror holder which in turn is pushed by a spring to a window at the top. The mirror holder contains eight holes in order to enable the fluid to circulate freely inside the chamber, even in the two-phase region. At the bottom of the chamber one finds an opening to a valve enabling the filling of the chamber. Viton O-rings close the SC.

Figure 3.8 The density-refractive index SCU.



The main purpose of this SC is to enable the determination of the density and the refractive index of various contained samples of  $\text{SF}_6$  of different densities. The density of each sample is calculated out of the corresponding mass and the total volume of the chamber. The mass is found by determining the difference in weight between a filled and an empty DER-SCU. Therefore, the chamber volume is as large as possible as the design allows. Taking into account the basic requirements pointed out in section 3.3.2 on page 39, the chamber is shaped as a cylinder of approximately 25 mm diameter and 37 mm height. The chamber volume is calculated instead of determined experimentally because the high accuracy with which all measures are known promises a higher accuracy this way. In fact, the calculation amounts to a volume of  $14404.4 \pm 3.5 \text{ mm}^3$  at  $48^\circ\text{C}$  [71].

The relative distance to the critical density of a contained sample is measured below CP in terms of the distance of the meniscus to the volumetric middle of the container (see section 2.4.3). The meniscus is imaged for each sample separately. In order to be able to calibrate the size of the image, on the side of the window adjacent to the fluid a circular marker of approximately 6.8 mm diameter is carved that is imaged together with the meniscus.

The refractive index is determined by measuring the difference in optical path between a filled and an empty DER-SCU of the distance from window to mirror inside the chamber. As will be discussed in chapter 5, this distance must be known to great precision. This has been taken care of by the use of an auxiliary piece while gluing the mirror to the mirror holder, so that the distance between mirror surface and top of the mirror holder is determined exactly by the length of this

piece which is 10.010 mm. The spring pushes the mirror holder to the quartz window keeping the mirror holder fixed w.r.t. the window. Still, changes of the distance from window to mirror are imaginable due to possible window distortion as a result of pressure changes or due to expansion of the mirror and the mirror holder as a result from changes in the temperature. By using a 0.5 inch thick window significant distortion is avoided as is evidenced by the interferograms. The choice of material for mirror and mirror holder enables to neglect expansion effects [71].