Operating parameters of liquid helium transfer lines used with continuous flow cryostats at low sample temperatures

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Abstract. Continuous flow cryostats are used to cool samples to a variable temperature level by evaporating a cryogen, e.g. liquid helium (LHe). For this purpose LHe is usually stored outside the cryostat in a mobile dewar and supplied through a transfer line. In general, the complete setup has to be characterised by the lowest possible consumption of LHe. Additionally, a minimum sample temperature can be favourable from an experimental point of view. The achievement of both requirements is determined by the respective cryostat design as well as by the transfer line. In the presented work operating data, e.g. the LHe consumption during cool-down and steady state, the minimum sample temperature, and the outlet quality are analysed to characterise the performance of a reference transfer line. In addition, an experimental transfer line with built-in pressure sensors has been commissioned to examine the pressure drop along the transfer line, too. During the tests LHe impurities occurred which restricted a steady operation.

1. Introduction

Continuous flow cryostats of varying geometries are widely used in cryogenic laboratories, see [1] and [2]. But all feature one common property, continuous flow cryostats have to be supplied steadily with the liquid cryogen i.e. the cooling agent to maintain stable operating conditions. The cryogen is supplied with a cryogenic transfer line which connects the cryostat with a mobile dewar. Within the cryostat the sample is cooled utilising the evaporation enthalpy or the cold gas enthalpy if temperatures above the respective boiling temperature are aspired. The performance of the cryostat setup is characterised by the consumption of cryogen and the minimum sample temperature. These properties are determined by the cryostat itself, but also considerably by the performance of the transfer line. Although, such cryogenic transfer lines are widely used in cryogenic laboratories, only little data about their heat leak, pressure drop, or outlet quality exists. Publications, like [3] and [4], cover the heat leak of transfer lines used in the context of cooling superconducting magnets or the heat leak and pressure drop of transfer lines used at decant stations, see [5]. In contradiction to these lines a rather high pressure drop along the transfer line for a continuous flow cryostat can be favourable since the pressure drop determines...
the operating temperatures if supplied with a two-phase flow. The heat leak is even more crucial considering the very low flow rate of just a few liters per hour.

The presented operating data provides the basis for the improvement of a single-channel LHe transfer line aiming lower consumption and operating temperatures. Decreasing the LHe consumption will reduce the operating costs and increase the experimental time, considering a fixed supply volume by the mobile dewar. Furthermore, decreased sample temperatures widen the field of application for continuous flow cryostats operated with LHe.

2. Experimental setup
The chosen experimental setup is similar to a standard continuous flow cryostat setup. To derive further information about the pressure drop along the transfer line, an experimental transfer line equipped with built-in pressure sensors was designed and commissioned. Thereby the basic design of the experimental transfer line was not modified leaving the hydraulic diameters and the insulation design unchanged. The experimental setup is shown in Figure 1.

![Figure 1. Experimental setup to characterise the transfer line; 1: mobile LHe Dewar, 2: transfer line, 3: continuous flow cryostat, 4: gas heater, 5: mass flow meter, 6: solenoid valve, 7: feed pump, 8: gas storage, 9: high vacuum pump.](image)

LHe is supplied via a mobile dewar (1) with a capacity of 100 l\textsubscript{LHe}. LHe is conveyed through the setup by the use of a feed pump (7) withdrawing LHe from the mobile dewar and pumping gaseous helium (GHe) into the low pressure gas storage (8). A flexible transfer line (2) without a cold gas return or liquid nitrogen shield connects the mobile dewar with the flow cryostat. The basic geometry of the transfer line features a riser length of 1060 mm, a horizontal length of 850 mm with a flexible length of 700 mm and a vertical length at the cryostat side of 410 mm. The transfer line consists of two concentric tubes with the inner tube conveying LHe and the
outer tube being the shell against the ambiance. The resulting radial gap is evacuated to
minimise the heat transfer by convection and residual gas conduction. Furthermore, several
layers (250 layers/cm) of multi layer insulation (MLI) are wrapped around the inner tube
reducing the radiation heat transfer.

In addition to a reference transfer line, an experimental transfer line with instrumentation
was examined, too. The first pressure sensor \( p_1 \) is located at the end of the riser section of
the experimental transfer line. The sensor \( p_2 \) is located after the needle valve and \( p_3 \) is measured
at the end of the horizontal section. The pressure measurement was realised with standard
pressure transmitters having a range of 0 to 0.1 \( \text{MPa}_{\text{abs}} \) with a typical measurement uncertainty
of 0.5 % FS. To compensate the cryogenic temperature level all pressure sensors are connected
to the inner tube of the transfer line via a capillary of 1.6 mm inner diameter. To minimise
systematic uncertainties caused by the bore a respective diameter of 0.5 mm was chosen. The
remaining deviation of the pressure signal was calculated to be below 20 Pa. In addition, a silicon
diode temperature sensor type DT-670A with an accuracy of \( \pm 0.25 \text{ K} \) at 4 K was positioned on
the inner valve body (\( T_v \)).

Within the continuous flow cryostat (3) the LHe evaporates, cooling the respective sample.
For the series of measurements a cryostat that is designed for low temperature microscopy
was used to simulate a common cryostat setup while examining the characteristic values of
the transfer line. The cryostat was equipped with an additional radiation shield cooled by the
returning cold GHe to compensate radiation heat transfer from the top lid. A top lid without
an optical feedthrough was used for this reason, too. The cryostat has been equipped with three
silicon diodes type DT-670A. The sensors were located at the inlet of the cryostat (\( T_{\text{in}} \)), at the
sample holder (\( T_{\text{sol}} \)) and at the outlet of the primary heat exchanger (\( T_{\text{hx}} \)). During operation
the cryostat is connected to a high vacuum pump (9). The helium leaves the cryostat after the
secondary heat exchanger where another pressure sensor (\( p_4 \)) is located. Afterwards the flow is
heated to ambient temperature by a heating appliance (4) before the flow rate is measured by a
mass flow meter (5) having an accuracy of \( \pm 0.004 \text{ g/s} \). Besides the needle valve the mass flow
rate can be also adjusted by a solenoid valve (6) located at the feed pump inlet.

3. Results and discussion

3.1. Cool-down behaviour

The cool-down data in Figure 2 shows that, starting from ambient temperature at around
300 K, the inlet and sample temperature approach the operating temperature within 40 min.
The systematic deviation of the inlet temperature from the sample temperature is caused by
the geometry of the inlet tube that constrains the sensor mounting. Due to the propagation
of the low temperature level along the setup the temperature of the primary heat exchanger
approaches the operating temperature within 50 min, indicating the beginning of the steady
operation. During steady operation at 4 K the valve body temperature \( T_v \) is around 12 K.

Due to the very low density of the cold gas at the beginning of the cool-down a maximum
mass flow rate between 0.01 and 0.05 g/s (0.3 to 1.4 L\(_{\text{LHe}}\)/h) is conveyed through the transfer
line within the first 39 min of the cool-down. During this time only a minor pressure drop along
the transfer line can be observed (see Figure 3). Due to the temperature drop after 39 min the
density increases which results in a sudden increase of the mass flow rate. At this point the mass
flow rate is adjusted by the needle valve to use the remaining cold gas enthalpy more effectively.

During the cool-down of the complete setup 1.5 L\(_{\text{LHe}}\) are withdrawn from the mobile dewar for
the case of the 4 K operation mode. The cool-down process could be more efficient by adjusting
the mass flow rate steadily so that all of the cold gas enthalpy is used. But this procedure
requires a very careful and time consuming process control [6].
3.2. Heat leak

The consumption of LHe for a steady state operation at very low temperatures is determined by the utilisable enthalpy difference between the inlet enthalpy and the saturated gas enthalpy. The enthalpy at the cryostat inlet is higher than the saturated liquid enthalpy due to the heat leak along the transfer line. Figure 4 shows the minimum sample temperature as a function of the actual mass flow rate. Since the mass flow rate is adjusted by the needle valve of the transfer line, the sample temperature decreases with lower mass flow rates due to the increased pressure drop across the needle valve.

**Figure 2.** Temperatures and mass flow rate during cool down of the test setup; $T_v$ ($\bigcirc$), $T_{in}$ ($\triangle$), $T_{sa}$ ($\square$), $T_{hx}$ ($\nabla$), mass flow rate ($\bullet$).

**Figure 3.** Pressure values during cool down of the test setup; $p_{dewar}$ ($\bigcirc$), $p_1$ ($\triangle$), $p_2$ ($\nabla$), $p_3$ ($\square$).

**Figure 4.** Minimum sample temperature in steady state ($\bigcirc$) as a function of the LHe mass flow rate valid for the reference transfer line.
It can be derived that the minimum sample temperature decreases gradually with lower mass flow rates. But below a mass flow rate of 0.07 g/s the temperature increases again. At and below this mass flow rate LHe evaporates completely within the transfer line. It can be derived from Figure 4 that a stable operation consumes 2 to 4 $l_{LHe}/h$, which is higher compared to a line with cold gas return consuming less than 1.0 $l_{LHe}/h$ at 4.2 K [1].

Since the outlet quality determines the actual cooling capacity, its experimental determination is crucial to characterise the examined transfer line. The outlet quality is determined by means of an electrical heater at the cryostat inlet. The power of the electrical heater is adjusted in a way that a temperature increase of the sample temperature is just not observed:

$$h_{out} = h_g(p = f(T_{sa}), x = 1) - \frac{P_{el,Heater}}{\dot{m}}$$

With an outlet quality of $x_{out} = 1$ the heat leak can be calculated using the mass flow rate and the defined inlet and outlet quality states, deriving the thermophysical properties from [7]. The heat leak of the reference transfer line without any instrumentation is determined to be $1.25 \pm 0.10$ W. At a mass flow rate of 0.14 g/s the quality is 0.45. A quality of 1 is observed at a mass flow rate of 0.07 g/s. In comparison, the heat leak of the experimental transfer line is determined to be $1.65 \pm 0.08$ W. For that line the outlet quality is determined to be 1 at 0.08 g/s and 0.55 for a mass flow rate of 0.14 g/s. The continuous flow cryostat’s heat leak is 1.8 W.

### 3.3. Pressure drop

The integration of pressure sensors along the test setup generates additional pressure drop data. The latter is important since the sample temperature is directly determined by the outlet pressure of the transfer line if the cryostat is supplied with two-phase helium. The pressure values for an operation at 4 K are shown in Figures 5 and 6.

![Figure 5. Local pressure values during operation at 4.0 K ($\dot{m} = 0.12$ g/s); $p_{dewar}$ ( ), $p_1$ (△), $p_2$ (▽), $p_3$ (□).](image5)

![Figure 6. Pressure drop during operation at 4.0 K; $p_{dewar} - p_1$ ( ), $p_1 - p_2$ (△), $p_2 - p_3$ (▽), $p_3 - p_4$ (□).](image6)

The results of the pressure measurement indicate that the largest fraction of the pressure drop along the transfer line is generated by the needle valve. Therefore, its proper adjustability is crucial for a steady cryostat operation. It can be derived from Figure 6 that the pressure drop...
along the cryostat is reasonable, too. Hence, the cryostat and the transfer line determine the overall conductance and by association the maximum mass flow rate.

3.4. Helium purity
In order to ensure a stable operation of the continuous flow cryostat a supply with pure helium is crucial. During the experiments LHe from two laboratory liquefiers was supplied resulting in very different behaviours of the test setup at temperatures below the normal boiling point of helium. Figure 7 shows the mass flow rates and the corresponding sample temperatures as a function of time.

![Figure 7](image)

**Figure 7.** Influence of the helium purity on the operating stability of a continuous flow cryostat setup; contaminated LHe: mass flow rate (○) and sample temperature (●), high-purity LHe: mass flow rate (□) and sample temperature (■).

If LHe with high purity is supplied, a steady operation at a stable sample temperature of 3.2 K is achieved at a mass flow rate of 0.09 g/s. In contradiction, starting at 0.09 g/s and at a temperature of 3.6 K the mass flow rate steadily decreases if LHe with a certain level of contamination is supplied. This is accompanied by an increasing pressure drop along the transfer line and a simultaneously decreasing sample temperature. If the mass flow rate approaches 0.07 g/s, where the transition between the two-phase and the cold gas region is observed, then the temperature starts rising. At a mass flow rate of 0.03 g/s the sample temperature is 9 K.

The observed mass flow rate decline is believed to be caused by a gradually clogging of the smallest cross section, i.e. the needle valve passage, due to the precipitation of solid hydrogen dissolved in LHe. This assumption is the result of a thorough fault analysis in which the cryostat and the transfer line itself could be excepted as a possible source of fault. Also solid particles in the LHe are probably not causing the observed behaviour since two filters were applied at the transfer line inlet allowing only particles with less than 35 µm or 0.2 µm to pass. Since the operation with saturated liquid nitrogen and GHe at around 6 K showed a similar behaviour to the pure LHe supply it was concluded that impurities are the most probable source of fault.

Figure 8 shows the theoretical limiting solubilities of hydrogen and neon in LHe as published in [8]. The limiting solubility $x_A = N_A / (N_A + N_{He})$ is given as mole fraction of component A, e.g. hydrogen or neon. Hydrogen as the most reasonable impurity in LHe has a limiting solubility of

![Figure 8](image)

**Figure 8.** Limiting solubilities $x_A$ of hydrogen (——) and neon (- - - -) in LHe as a function of temperature according to Jewell and McClintock [8].
$x_A = 10^{-10}$ or 0.1 ppb at 4.2 K. To quantify the amount of solid hydrogen dissolved in LHe some assumptions were made. First, it is stated that the complete pressure drop between the mobile dewar and the sample holder is generated by the needle valve. Secondly, it is assumed that the pressure drop is equivalent to an isenthalpic change of state. It follows that the temperature at the needle valve is reduced from 4.2 K to 3.2 K decreasing the limiting solubility to $x_A = 10^{-14}$ or 0.01 ppt. The difference in the limiting solubilities results in a precipitation of solid hydrogen that will most likely sediment at the angle valve, narrowing the needle valve passage whose equivalent diameter is 0.7 mm.

In addition, the pressure drop across the needle valve generates a reasonable amount of helium vapour. Since solid hydrogen is not solvable in GHe, the respective hydrogen will precipitate, too. Due to the isenthalpic throttling from 0.0992 to 0.0320 MPa a quality of $x = 0.17$ is generated by the needle valve. Considering both mechanisms of precipitation $3.2 \times 10^{-10} \text{ g}$ or $4.2 \times 10^{-6} \text{ mm}^3$ hydrogen may sediment at the needle valve per minute. Finally, it must be stated that the limiting solubilities may vary by several orders of magnitude since they are calculated assuming ideal dissolving parameters [8]. Since there exists no experimental data, this is the most suitable approach to quantify and explain the observed behaviour.

4. Conclusion
A test setup featuring a continuous flow cryostat was used to characterise a single-channel transfer line regarding its heat leak, quality and pressure drop. During the experiments it was found that the purity level of the supplied LHe does influence the steady operation of the cryostat setup. Derived from a systematic fault analysis a clogging of the needle valve passage by precipitating solid hydrogen was found to be the most probable cause for that behaviour. Since the setup is very sensitive to LHe impurities, it is also a reliable indicator of such impurities.

As a result of the analysis some improvements of the transfer line design can be suggested. First, the comparatively high temperature of the needle valve body indicates some potential to minimise the heat leak. Secondly, the layer density of the transfer line may be reduced, to minimise the heat leak due to conduction along the MLI. Since the largest pressure drop fraction is generated by the needle valve, it seems advantageous to distribute the pressure drop over the complete length of the transfer line. This would result in a better adjustability of the mass flow rate. It might also reduce the transfer line’s susceptibility to clogging caused by LHe impurities since the needle valve could be opened further, widening the smallest cross section.

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