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Thermal Parameters of a Sealed Lambda-Point Cell Developed at PTB

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1. Introduction

In the temperature range below the triple point of equilibrium hydrogen at 13.8033 K, the ITS-90 contains only the helium vapour-pressure scales as quasi-continuous fixed points. But the realisation of these scales requires measuring the vapour pressure. Furthermore, superconductive reference samples can be used as transfer standards for comparing temperature scales. In general, these samples have to be calibrated individually. To improve this situation, the lambda transition of liquid ^{4}He has been proposed to be an excellent alternative for realising a temperature fixed point using sealed cells, i.e. without measuring other physical quantities than temperature [1-3]. At PTB sealed lambda-point cells (SLPCs) have been developed as fixed-point devices [4], which are robust and compact, very easy in use, and which ensure a repeatability of the lambda-transition temperature that is limited only by the stability and resolution of the thermometer used for the detection of the transition. In this document, the thermal parameters of one of the SLPCs are described. These parameters have been determined in detail in order to provide simple instructions for a fixed-point realisation at the highest level of accuracy under experimental conditions being accessible to most of the potential users. A comparison with other realisations of the lambda transition will be performed later on.

2. Operating principle of the SLPC

On cooling, liquid ^{4}He undergoes a second-order phase transition, the so-called lambda transition, from the normal-fluid state to the superfluid state at a temperature T_λ of 2.1768 K according to ITS-90. (The ITS-90 transition-temperature value is based on a realisation using an open system described in [5].) This phase transition is accomplished by a drastic rise of the thermal conductivity in the superfluid state by many orders of magnitude. Thus, along a column of liquid ^{4}He carrying a heat flux, large thermal gradients occur only if the column has at least partly a temperature being larger than T_λ. This appearance of thermal gradients is used for detecting the lambda transition and, consequently, measuring T_λ. In a temperature range of several millikelvins below and above T_λ, the heat capacity of both phases is largely enhanced. Even though the phase transition is of second order, this enhanced heat capacity of liquid ^{4}He near T_λ enables to realise the transition as a fixed point under practical experimental conditions because it minimises the influence of parasitic heat flows.

Since more than 30 years the lambda transition of ^{4}He has been widely studied experimentally as well as theoretically as a model phase transition. It has been found that the lambda transition appears to be remarkably insensitive to external influences. The following main effects are known to affect T_λ [2]:

- T_λ is depressed by pressure at a rate $\mathrm{d}T_\lambda/\mathrm{d}p$ = -87.8 nK/Pa, leading to a depression by a liquid column of height h at a rate $\mathrm{d}T_\lambda/\mathrm{d}h$ = 1.27 µK/cm.

- T_λ is depressed by ^{3}He, the only remaining impurity in liquid ^{4}He, at a rate $\mathrm{d}T_\lambda/\mathrm{d}X$ = -1.45 K, where X is the molar fraction of diluted ^{3}He.

- For heat fluxes $Q \leq 10$ µW/cm^2, T_λ is depressed by the heat flux approximately according to the formula $\Delta T_\lambda / T_\lambda = (Q/Q_0)^\alpha$, where $\alpha = 0.813$ and Q_0 = 570 W/cm^2. For larger heat fluxes, the depression may follow another formula, but this has not yet been investigated in detail.

Choosing an appropriate experimental design for realising the lambda transition, the corrections, which have to be applied to T_λ, amount only to several microkelvins according to the formulas given above.

3. Design of the PTB-type SLPCs

At PTB, two models of SLPCs have been designed and built. Their designs meet the following demands. First, the SLPCs have to be simple, compact and robust. Second, all thermal parameters being necessary for realising the lambda transition and, therefore, for determining T_λ should be easily measurable or calculable. Third, it should be possible to realise the lambda transition for thermometer calibrations with a stability within a few microkelvins over very long periods of time, which are in principle only limited by the running time of the cryostat. Forth, to enable a wide application, the fixed-point realisation should not require complicated experimental arrangements including high-precision temperature control. Fifth, small heat fluxes have to be used for detecting the transition, i.e. the dimensions of the SLPCs should be optimised in such a way that the power applied during the realisation of the lambda transition does not drive the superfluid phase far away in a region, in which dissipative effects appear and which may be physically not well understood. For the SLPCs of PTB design, the corrections to be applied to the T_λ values are of order of only a few ten microkelvins, i.e. very small.

The small PTB-type SLPC consists of a cylindrical main body and a lower copper plate, which are connected by a hard-soldered stainless-steel tube of 6 mm outer diameter, 5 mm inner diameter and 30 mm whole length (see Figure 1). The stainless-steel tube is the so-called delay line. The main body of the SLPC contains two holes of 5.2 mm diameter for rhodium-iron resistance thermometers (RIRTs) of standard type and two holes of 3.2 mm diameter for germanium resistance thermometers (GRTs) or other semiconducting thermometers. A thin stainless-steel capillary (outer diameter 3 mm) serves as a thermal link to the bath temperature of the cryostat. It is screwed into the top of the main body and hard soldered into a copper flange, which can be screwed to the bottom of the thermal bath of the cryostat. The SLPC (inner volume about 10 cm^3) has been filled trough a copper capillary of 2 mm diameter with high-grade ^{4}He (purity 99.9999%). The copper capillary has been pinched off at a pressure of approximately 70 bar. The maximum length of the whole assembly is 93 mm and the maximum diameter is 35 mm, i.e. it can be applied in dipstick cryostats. A heater is attached to the lower copper plate. For adjusting the plate temperature and thus the heating power, a thermometer can be screwed to this plate. The stainless-steel tube of the delay line carries less than 10 % of the heating power at temperatures near T_λ.

The outer dimensions of the large PTB-type SLPC are: diameter 60 mm and length 100 mm. The delay line has the same dimensions as for the small SLPC. A prototype of this SLPC model has been already built. It will be tested in near future.

4. Operation of the small PTB-type SLPC

The SLPC is operated in the following way. The copper flange of the thermal link is connected to the thermal bath of a cryostat that has a temperature T_0 below the temperature $T_\lambda = 2.1768$ K of the superfluid to normal-fluid transition. The temperature T_0 is stabilised at a level of ±1 mK. Monitoring the temperature readings of the thermometers in the main body of the SLPC during cooling, one can find a small plateau in their time dependence indicating the condensation of the ^{4}He gas into the liquid state. After one has cooled the whole SLPC to temperatures below T_λ, there is no measurable temperature difference between the main body and the lower copper plate because of the extremely high thermal conductivity of the superfluid ^{4}He. Turning on the current through the heater one rises the temperature of the lower copper plate above T_λ. Adjusting the heating power, one can fix the position of the interface between the normal-fluid ^{4}He in the lower part and the superfluid ^{4}He in the upper part at the desired height in the stainless-steel tube. In this configuration, the temperature of the main body of the SLPC and hence the temperature of the thermometers inside the holes of the main body is the measured value of T_λ. This value of T_λ has to be corrected for the depression by the used heat flux, the height of the liquid column above the phase boundary and the temperature difference between the superfluid ^{4}He and the main body. Because of the large heat capacity of the superfluid ^{4}He and the high thermal resistance to the bath temperature T_0, the main body of the SLPC can be held at T_λ over very long periods being only limited by the running time of the cryostat.

The experiments were carried out in a ^{3}He-dipstick cryostat inside a commercial 100 l helium transport vessel. The copper flange of the thermal link of the SLPC was screwed to the ^{3}He pot, the temperature T_0 of which was stabilised slightly below T_λ by means of a temperature controller and by pumping the 1K pot of the dipstick cryostat. The temperatures of the main body and the lower copper plate of the SLPC, respectively, were measured by means of two GRTs, having a high sensitivity, and two resistance bridges, the data of which were collected by a PC. The resolution of the temperature measurement was about 1 μK, which enabled a very sensitive investigation of the thermal conditions. The temperature reading of the lower GRT attached to the copper plate served as an indicator for the change of the height of the normal-fluid column in the lower part of the stainless-steel tube. The heating power was supplied to the lower copper plate by a high-stability source meter. The reading of the upper GRT, located in one of the holes of the main body of the SLPC, yielded the information on the transition temperature T_λ.

5. Experimental results

During the investigation of the small PTB-type SLPC, 15 cooldowns were carried out with the ^{3}He-dipstick cryostat. The cooldowns from room temperature to liquid helium temperature and the warm-ups were done very fast in short periods (1 h to 2 h). Nevertheless, the maximum difference of all uncorrected temperature values measured with the upper GRT at the lambda transition, realised under different conditions (heat flux, height of the superfluid column), was 80 μK. The values for T_λ determined during different realisations in two subsequent cooldowns coincide within ± 7 μK after correcting for the heat flux depression, the hydrostatic head above the phase boundary and the temperature difference at the thermal resistance between the superfluid ^{4}He and the main body of the SLPC.

Figure 2 shows an example for the stability of the temperature reading of the upper GRT over a period of more than 5 hours. The reading T_λ is drawn relative to the temperature value $T_{\lambda 0}$ obtained by extrapolation to zero heat flux. The heating power was adjusted simply by hand looking at the reading of the lower GRT attached to the bottom copper plate. While the reading of the lower GRT was adjusted within a few millikelvins, the temperature of the upper GRT was stable within a few microkelvins. The longest plateau for a T_λ measurement during the performed experiments lasted over 18 hours. These results show that the SLPC can be easily used under conditions being accessible to most of the users.

Since the realised transition temperature depends on the heat flux, a careful investigation of the thermal parameters of the SLPC was carried out. The thermal resistance of the thermal link from the main body of the SLPC to the bath temperature of the cryostat via the stainless-steel capillary was found to be 2.5×10^4 K/W near T_λ. Changing the bath temperature of the cryostat, the dependence of T_λ on the heat flux has been determined over a range from 2 μW/cm^2 to 86 μW/cm^2. An example for the decrease of T_λ with stepwise increasing heat flux is given in Figure 3. The relative depression of T_λ in dependence on the heat flux is shown in Figure 4 for different realisations of the lambda transition during different cooldowns. A numerical approximation to literature data is shown also. The form of the approximation function was suggested by theoretical considerations of phase transitions [6]. A comparable dependence of $\Delta T_\lambda/T_\lambda$ on the heat flux was found in [7]. For a single cooldown, the extrapolation of T_λ to zero heat flux is illustrated in Figure 5. The necessary correction to T_λ due to the heat flux, as can be clearly seen, amounts to less than 20 μK for the maximum heat flux used.

The uncertainty budget for the comparison of transition temperatures T_λ obtained under different experimental conditions is given in Table 1. The main uncertainty component results from the instability of the GRTs during the fast thermal cycling between room and helium temperatures. Its estimate is based on the comparison of the readings of both GRTs below the lambda transition that has been performed prior to realising each transition. This uncertainty component may be reduced by using RIRTs, but for the investigation of the thermal parameters of the SLPC, the high sensitivity and the small time constant of the GRTs were most important. The heat-flux correction has been applied in each case using a linear extrapolation as illustrated in Figure 5. The height of the superfluid column, which is necessary for calculating the head correction, has been estimated in the following way. Considering the heat flow both in the ^{4}He and in the stainless-steel tube, the difference between the readings of the two GRTs and the heating power yield the thermal resistance of the normal-fluid ^{4}He in the lower part of the stainless-steel tube. Using literature data for the thermal conductivity of normal-fluid ^{4}He, the height of the normal-fluid column can be calculated from this thermal resistance. The superfluid column ranges from the phase boundary at the upper end of the normal-fluid column to the upper surface of the liquid ^{4}He.

Table 1: Uncertainty budget for the comparison of transition temperatures T_λ obtained under different experimental conditions.

Type B uncertainty components (mK)	
1. Heat-flux correction	0.005
2. Head correction	0.002
3. Temperature difference between the SLPC main body and the superfluid ^{4}He	0.005
4. Drift correction	0.001
5. Resistance measurement	0.005
6. Temperature difference between the GRT sensor element and the SLPC main body	0.005
7. Thermometer instability	0.045
Type B combined (mK)	0.046
Type A uncertainty component (mK)	0.010
Standard combined uncertainty (mK)	0.047

Considering the estimated uncertainty, the obtained differences between the corrected values for the lambda-transition temperature obtained under different experimental conditions are not significant. In view of the small estimates for the uncertainty components caused by the SLPC itself, the true non-repeatability seems to amount at most about 10 µK. This will be checked soon using RIRTs. Furthermore, it is planned to compare the tested SLPC with other SLPCs in order to check the influences of the thermal conditions and the purity of the gas sample.

6. Conclusions

Using the SLPC developed and investigated at PTB, it is possible to realise the lambda transition of ^{4}He as a low-temperature fixed point at the highest level of accuracy. The non-repeatability of the transition temperature amounts to at most a few 10 µK. This conclusion is limited only by the instability of the applied GRTs. The thermal parameters of the SLPC have been investigated in detail. These parameters meet all demands on a high-precision, but easy to use fixed-point devices. The small PTB-type SLPC is of compact design and can be applied in dipstick cryostats. The realisation of the lambda transition does not require complicated experimental arrangements including high-precision temperature control. Thus, the SLPC can be used both as fixed-point device in metrology and as transfer standard for the dissemination of the temperature scale under conditions, which are accessible to most of the users in low-temperature laboratories.

Acknowledgements

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Figures

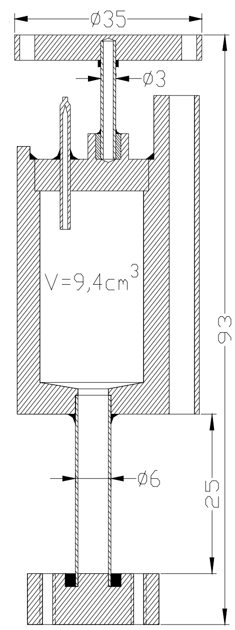

Figure 1: Schematic diagram of the small PTB-type SLPC

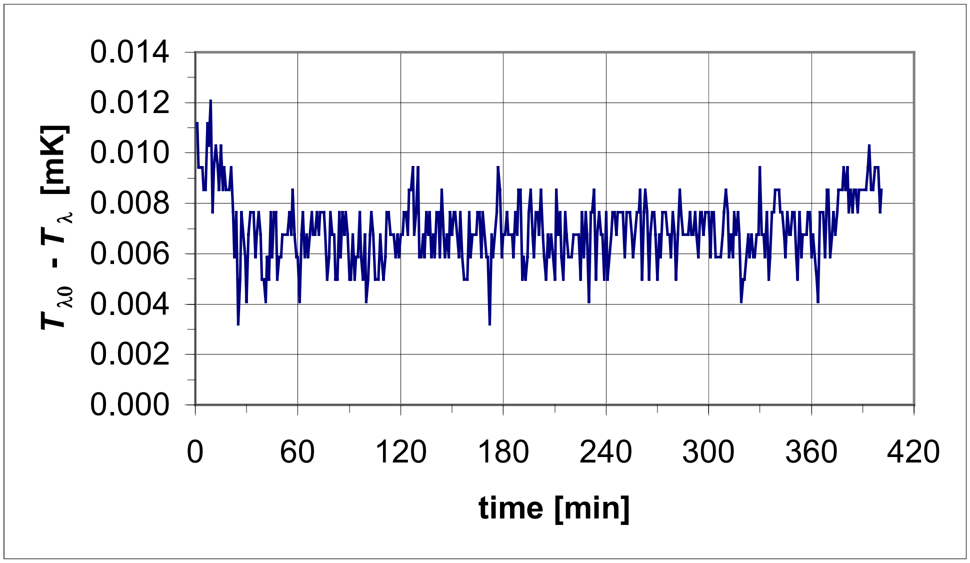

Figure2: Stability of the temperature reading of the upper GRT in the main body of the SLPC that detects the temperature of the lambda transition.

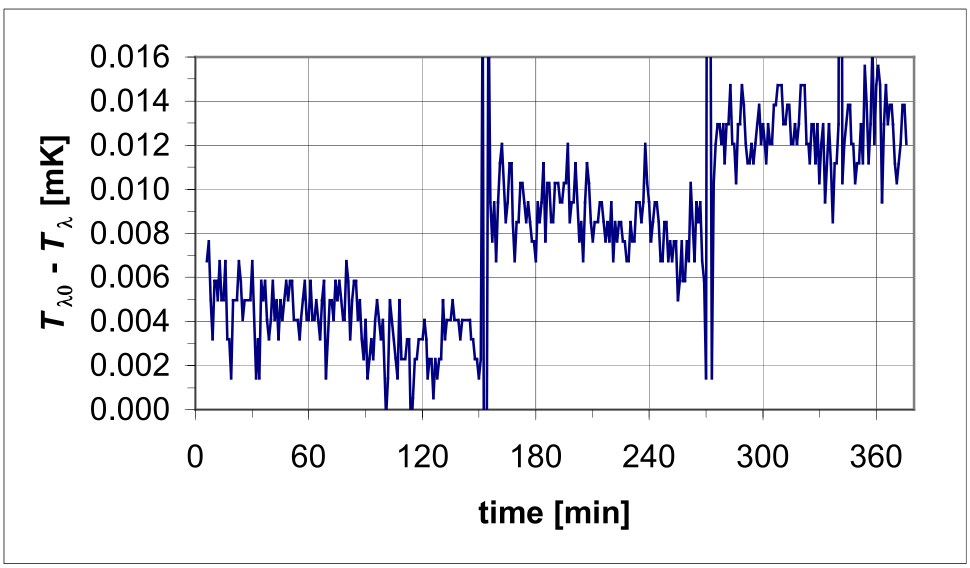

Figure 3: Depression of T_λ by the heat flux through the normal-fluid to superfluid interface. The heat flux was increased from 21.0 μW/cm^2 to 71.5 μW/cm^2 in two steps of 26.4 μW/cm^2 and 24.1 μW/cm^2.

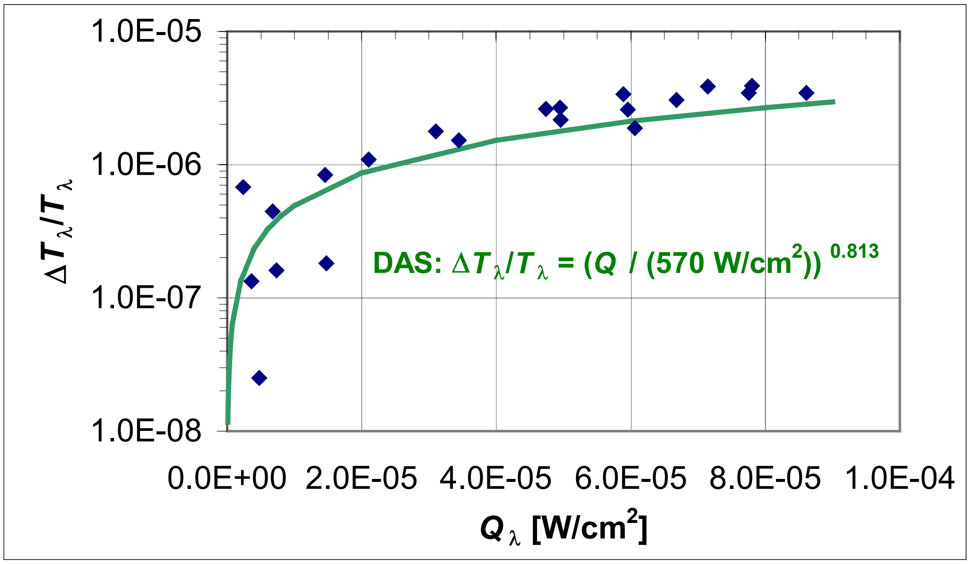

Figure 4: Experimental data for the relative depression of T_λ by the heat flux through the normal-fluid to superfluid interface. The curve DAS represents a fit to the data given in [4].

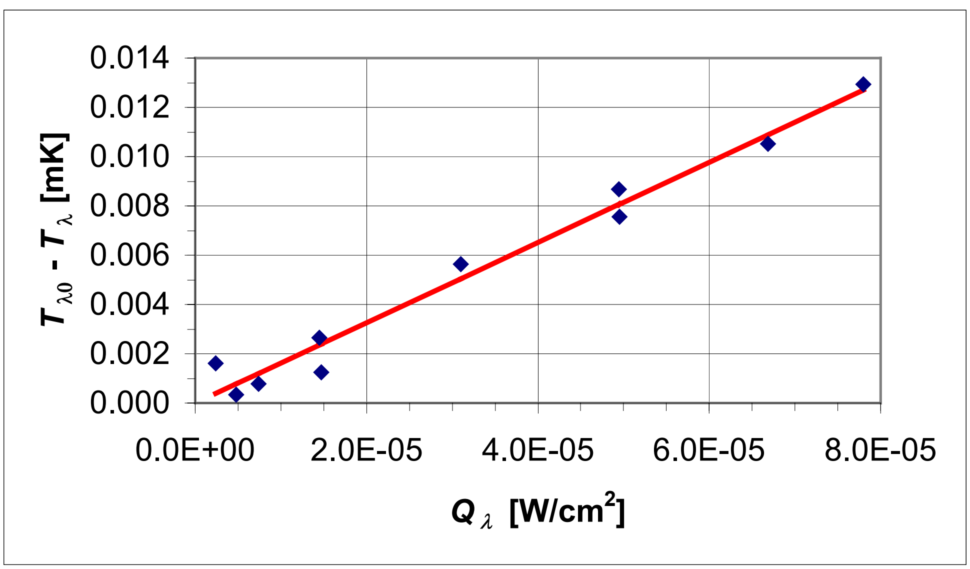

Figure 5: Experimental data for T_λ measured using different heat fluxes through the normal-fluid to superfluid interface. The solid line results from a linear fit to the data. It is used to determine T_λ for zero heat flux.