DESY SUMMER STUDENT PROJECT

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Characterization of the Cryo-con cryocooler at the P02.1 beamline

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Photon Physics, Low Temperature Physics

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Short description of the research project

This project deals with characterization of the Cryo-con cryocooler available to users at the P02.1 beamline. It contains practical information about the consumption of liquid nitrogen and filling procedure. Furthermore the method of refinement of the PID parameters is described. The last part of the report presents measurements of temperature gradients in vicinity of the cryocoolers' nozzle.

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Contents

1 Introduction

The cryocooler from the Cryo-Systems enables P02.1 users to perform in-situ X-ray diffraction (XRD) experiments in the temperature range from 90 K to approximately 400 K. Build in controller makes possible experiments under different temperature conditions. This work focuses on describing the performance and properties of cryocooler available to users at the P02.1 beamline.

2 Goals of the research project

The main goal of this project is to investigate various aspects relevant for future in-situ XRD low temperature experiments conducted with the Cryo-con cryocooler. Particular goals of this project can be divided in to three main parts:

- determine consumption of liquid nitrogen (LN_2),
- refine controller's PID parameters,
- map temperature gradients in the vicinity of the cryocooler nozzle.

3 Short introduction to the Cryo-con cryocooler

3.1 Description of cryocooler

The cryocoolers are generally used for achieving temperatures which are usually below the ambient temperature and above the boiling point of nitrogen (77.2 K) or helium (4.2 K). The set up at the P02.1 beamline (see Figure 1) consists of four main parts: cryocooler's dewar, storage dewar (see Fig.2 and Fig.3), transfer line with the nozzle (see Fig.4) and the panel with controllers (see Fig.5). Next chapters give more detailed description of respective parts. It should be noted here that more details can be found inside the manufacturer's manual.

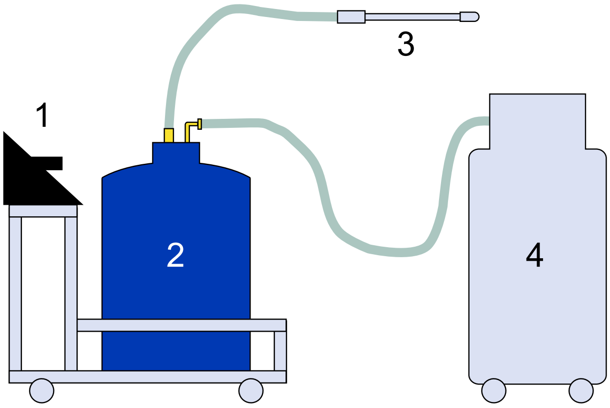

Figure 1: Main parts of the cryocooler: (1) controller panel, (2) dewar, (3) transfer line with the nozzle and (4) storage dewar.

3.1.1 The cryocooler's dewar

The dewar has two liquid nitrogen reservoirs (see Fig.2 and Fig.3). The inner reservoir is surrounded by the main outer storage reservoir as it is illustrated in Fig.2. The DC voltage of 24 V controls the solenoid valve that is located inside the dewar. This solenoid is used when automatically filling the inner liquid nitrogen reservoir (see (4) in Figure 5). The heater assembly in the inner reservoir vaporizes liquid nitrogen to provide the cold gaseous stream. The flow rate is controlled by external DC power supply. The twin banana plug for this supply is located on the right side of the digital flow control panel (see (3) in Figure 5). When the power to the reservoir heater is turned off, the flow of gas stream slowly stops. In such a scenario N_2 gas will become very cold ($\sim$ 83 K) and stable, without requiring control. The cryocooler's dewar needs to be periodically refilled from the transport liquid nitrogen dewar as it is shown in Fig.1(see (4)). Standard transport liguid nitrogen dewars have usually two valve outlet ports: one for liquid removal and another one for gas removal. One should avoid touching liquid or cold metal surfaces with bare skin. The liquid nitrogen stored in these containers is

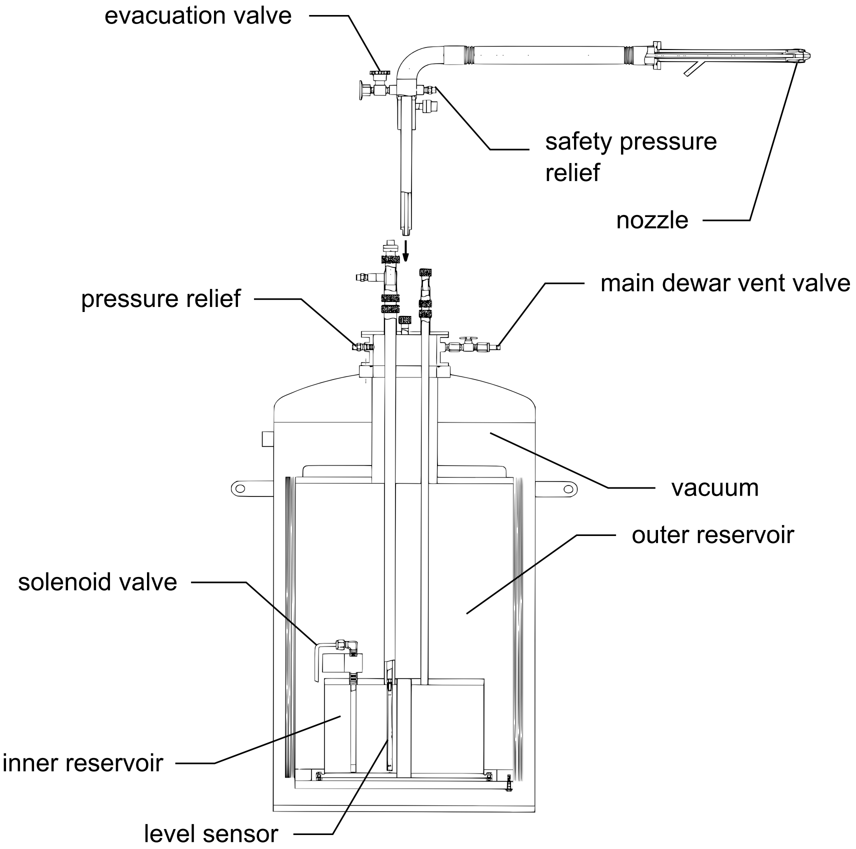

Figure 2: The main components of the cryocooler system.

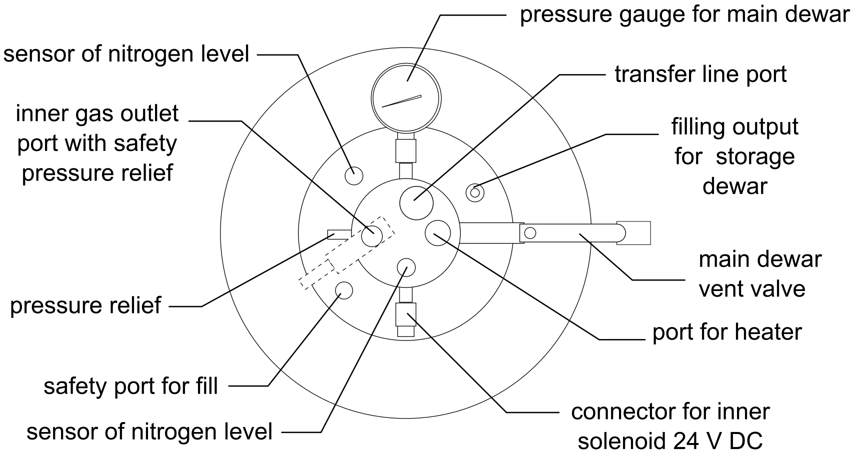

Figure 3: The top view on the cryocooler's dewar.

extremely cold: 77.2 K (-195.8 °C). Before the first filling of the cryocooler's dewar it is recommended to purge the transfer lines and reservoirs with flow of nitrogen gas. The purpose of purging is to remove air and moisture from the system and thus prevent any ice formation inside the cryocooler [1].

3.1.2 Transfer line with the nozzle

The vacuum insulated transfer line provides lowest terminal temperatures to the delivered cold flow. Resistive heat exchanger is used to adjust the temperature of outgoing gaseous N_2 stream. A platinum temperature sensor (PT100) is installed inside the nozzle behind the heat exchanger and measures the gas temperature just before leaving the tip. The temperature controller will use this sensor to automatically control any gas temperature selected (control loop 1). Furthermore the platinum sensor is wired in a 4-wired configuration in order to eliminate any error due to lead resistance. Another heat exchanger with the resistive heater is installed at the nozzle tip. The purpose of this second heater is to keep the tip of cryocooler ice-free. The Cryo-con controller maintains the temperature of the tip constant at 295 K (control loop 2). To provide an ice an ice-free environment at low temperatures, the nozzle is heated by copper wire in tip. Shield gas is not needed. The manufacturer affirms that cryogenic insulating techniques allow us to deliver nitrogen gas to the tip at temperatures down to 80 K [1].

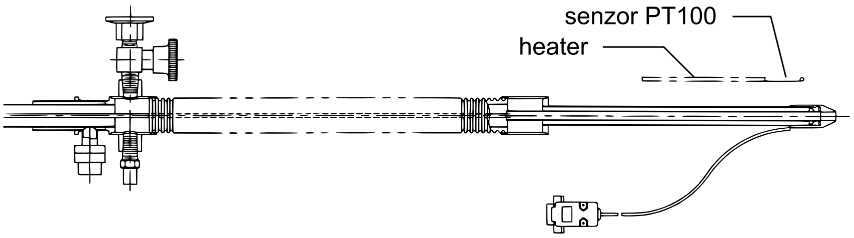

Figure 4: Detailed view of the transfer line with the nozzle.

3.1.3 Controller panel

Both liquid nitrogen reservoirs are monitored electronically (see (1) in Fig.5). Liquid levels are displayed continuously. Re-filling of inner nitrogen reservoir is fully automatic. The outer reservoir has to be refilled manually. However such refilling can be done without disturbing the experiment thus providing an unlimited operating time. The full range of the Channel 1 (inner reservoir) sensor is 18 cm. For the Channel 2 (outer reservoir) sensor it is 54.6 cm. The alarm low limits for the Channel 1 and 2 are set to 3.4 cm and 10 cm, respectively.

The controller allows a different user's adjustments. One of the main goals of this project was focused on refinement of PID parameters. When pressing the button for the PID table on the temperature controller its display shows three editable rows (see Fig.6). First row is for choosing number of editing table, second one *N* presents the number of set points, which are in selected table, and the third one is for editing. In the end press the button *Save Table & Exit* and after that button Loop1. One should not forget to change type (table) and rewrite table number. To activate the control loop one should press the button *Control*.

Figure 5: Controller panel – (1) liquid nitrogen level monitor, (2) temperature controller, (3) heater regulator and (4) plug for solenoid (24 V DC).

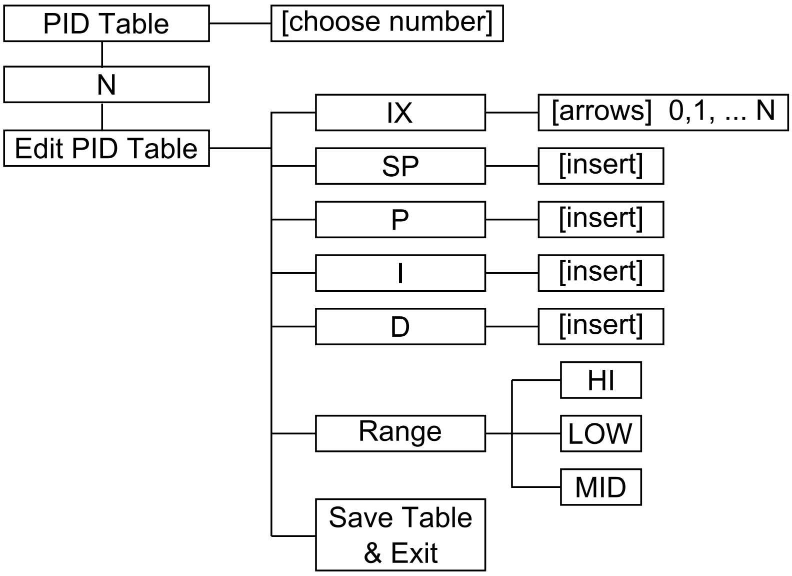

Figure 6: Scheme of the PID settings for temperature controller.

Shutting down the cryocooler:

- **short term** – reduce the flow heater to a very small setting (∼ 1 to 2 V), the objective is to keep a minimum flow rate to maintain a positive pressure in order to prevent moisture for entering the nozzle,
- **long term** – turn off the power to the vaporized heater and place a rubber stopper into the inner nozzle tip, to seal against moisture [1].

4 PID parameters

Proportional-Integral-Derivative (PID) control is the most common control algorithm has been universally accepted in industrial control. The popularity of PID controllers can be attributed partly to their robust performance in a wide range of operating conditions and partly to their functional simplicity, which allows engineers to operate them in a simple, straightforward manner.

As the name suggests, PID algorithm consists of three basic coefficients; proportional, integral and derivative which are varied to get desired response. In a typical control system, the process variable is the system parameter that needs to be controlled, such as temperature (°C), pressure (psi), or flow rate (liters/minute). A sensor is used to measure the process variable and provide feedback to the control system. The control design process begins by defining the performance requirements. Control system performance is often measured by applying a step function as the set point command variable, and then measuring the response of the process variable. Commonly, the response is quantified by measuring defined waveform characteristics [2].

4.1 P – parameter

The proportional component depends only on the difference between the set point and the process variable. This difference is referred to as the error term. The proportional gain (K_c) determines the ratio of output response to the error signal. For instance, if the error term has a magnitude of 10, a proportional gain of 5 would produce a proportional response of 50. In general, increasing the proportional gain will increase the speed of the control system response. However, if the proportional gain is too large, the process variable will begin to oscillate. If K_c is increased further, the oscillations will become larger and the system will become unstable and may even oscillate out of control [2].

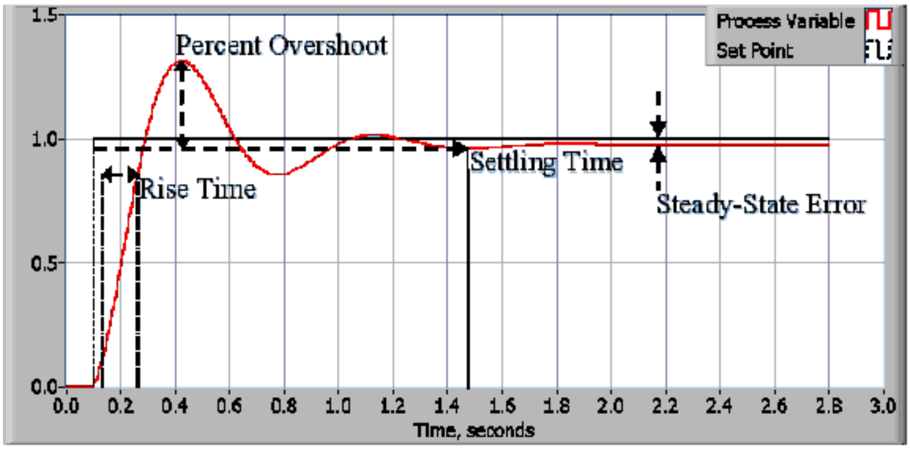

Figure 7: Response of a typical PID closed loop system.

4.2 I – parameter

The integral component sums the error term over time. The result is that even a small error term will cause the integral component to increase slowly. The integral response will continually increase over time unless the error is zero, so the effect is to drive the steady-state error to zero. The steady-state error is the final difference between the process variable and the set point. A phenomenon called integral windup results when integral action saturates a controller without the controller driving the error signal toward zero [2].

4.3 D – parameter

The derivative component causes the output to decrease if the process variable is increasing rapidly. The derivative response is proportional to the rate of change of the process variable. Increasing the derivative time (T_c) parameter will cause the control system to react more strongly to changes in the error term and will increase the speed of the overall control system response. Most practical control systems use very small derivative time (T_c), because the derivative response is highly sensitive to noise in the process variable signal. If the sensor feedback signal is noisy or if the control loop rate is too slow, the derivative response can make the control system unstable [2].

4.4 Tuning

The process of setting the optimal gains for P, I and D to get an ideal response from a control system is called tuning. There are different methods of tuning - for example guess and check method or the Ziegler Nichols method.

The Ziegler-Nichols method is another popular method of tuning a PID controller. It is very similar to the trial and error method wherein I and D are set to zero and P is increased until the loop starts to oscillate. Once oscillation starts, the critical gain K_c and the period of oscillations T_c are noted. The P, I and D are then adjusted as per the tabular column shown below [2].

Control	P	I	D
P	0.5 K_c	-	-
PI	0.45 K_c	0.83 T_c	-
PID	0.60 K_c	0.5 T_c	0.125 T_c

Table 1: Ziegler-Nichols tuning, using the oscillation method.

5 Experimental part

5.1 Consumption of liquid nitrogen

One goal of our work was to determine consumption of liquid nitrogen. We used a system of network camera AXIS in the hutch P02.1, which sends images of controller panel each minute. In this way we recorded change in the level sensors as a function of time. Extracted data were put into graphs and fitted with linear dependence (see Fig.8). Results for different temperatures (150 K, 200 K, 250 K and 295 K) show that the value of slope is same for all temperatures. The consumption is 0.014 cm/min (0.84 cm/hour). It should be noted here that the DC voltage on internal vaporizer which controls the flow of gaseous N_2 was set to 22 V. It was kept constant for other experiments as well.

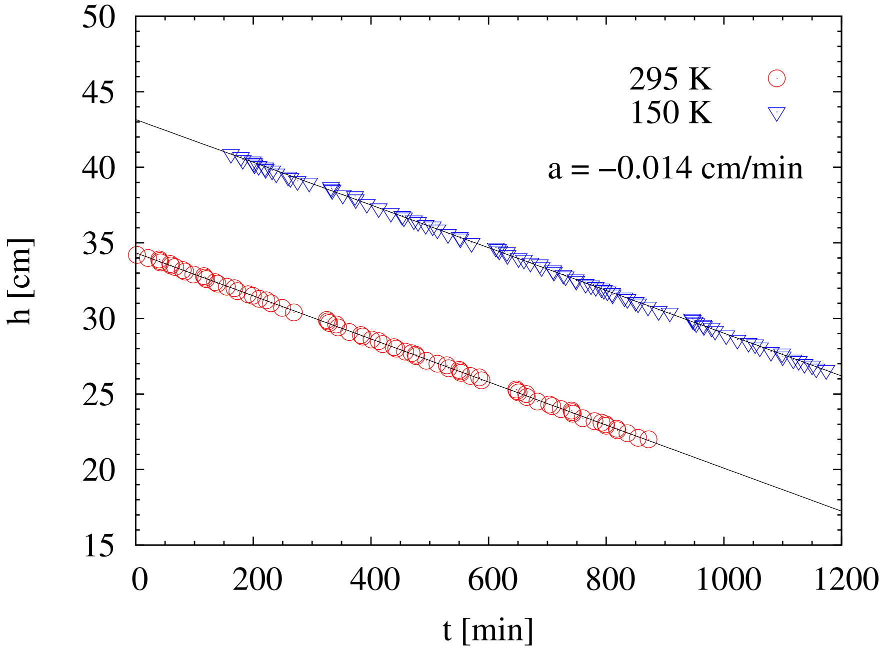

Figure 8: Consumption of nitrogen for different temperatures (22 V).

Working with the cryocooler helps to get an idea about the optimal filling procedure. It is recommended for users to fill the main dewar of the cryocooler as it is described in the following table (Tab.2).

Status	**Vaporizer voltage** [V]	**LN_2 filling**	**Notes**
active	22	twice per day	-
inactive	3	once per two days	switch off heater (Loop 1)

Table 2: Recommended procedures for filling the outer reservoir of the cryocooler.

5.2 Refining of PID parameters

As it was described in previous sections, the tuning of PID parameters is used for setting the optimal gains for P, I and D in order to get an ideal response from a control system. We were searching for the best PID parameters in range from 300 K to 90 K. Tests were made for both processes - cooling (from 300 K to 90 K with step 10 T) and heating (from 95 K to 295 K with step 10 K). Everything started with setting of PID parameters as it is shown in the Fig.6. Obtained PID table (see Table 5 in the manufacturer's manual [1]) consists of 8 set points: 295, 280, 250, 220, 190, 160, 130 and 100 K. Using the Ziegler-Nichols method the P parameter was tuned, whereas the I and D parameters were set to zero. The value of P parameter was refined for each set point. The ideal value of the P parameter can be obtained when process variable (in our case temperature) oscillates with a constant amplitude. Then one needs to calculate the period of oscillations. Obtained period T_c is then according to the Ziegler-Nichols method used to calculate the I and D parameters (take a look on table 1, page 7). In this work the Temperature control program (written in Python) was used [3]. Figure 9 shows an example of the temperature profile when controlling the cryocooler with optimal set of PID parameters (listed in Tab.3).

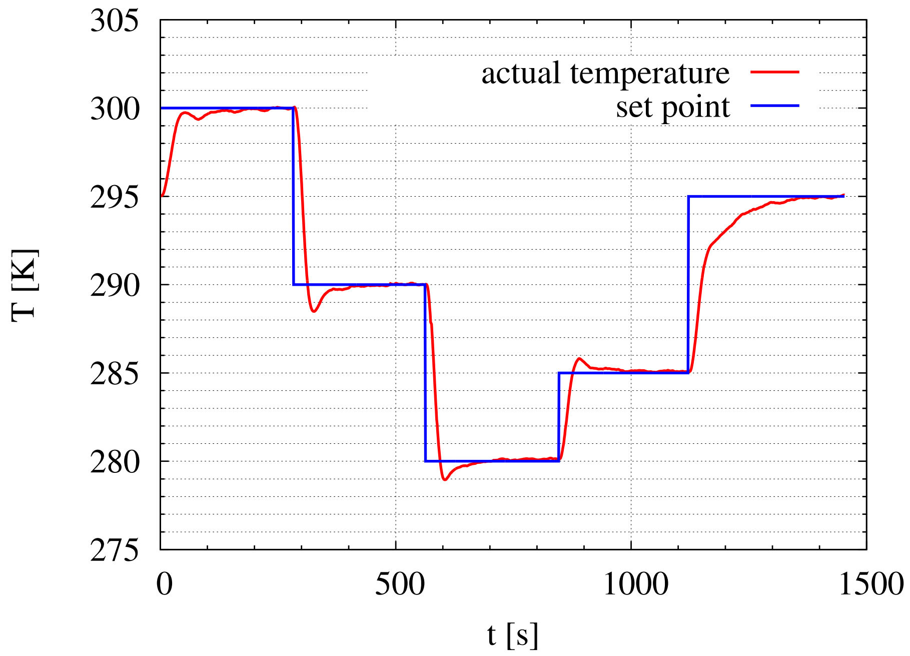

Figure 9: The temperature profile when controlling the cryocooler with optimal set of PID parameters.

Set point [K]	**P**	**I**	**D**
295	3.0	60.0	6.0
280	3.0	65.0	6.0
160	4.2	22.0	1.0
130	4.5	20.0	1.0
100	5.0	20.0	1.0

Table 3: Optimalized set of the PID parameters.

5.3 *Temperature gradients around the nozzle*

The last investigations were addressing temperature gradients around the nozzle. To check the temperature the sample usually experience when exposed to the cold N_2 stream, an external PT100 temperature sensor was used. The PT100 was placed in front of the nozzle and fixed with an independent stand. The nozzle could be moved along three main axes x, y and z. Figure 4 shows how the PT100 was positioned with respect to the cryocooler's nozzle.

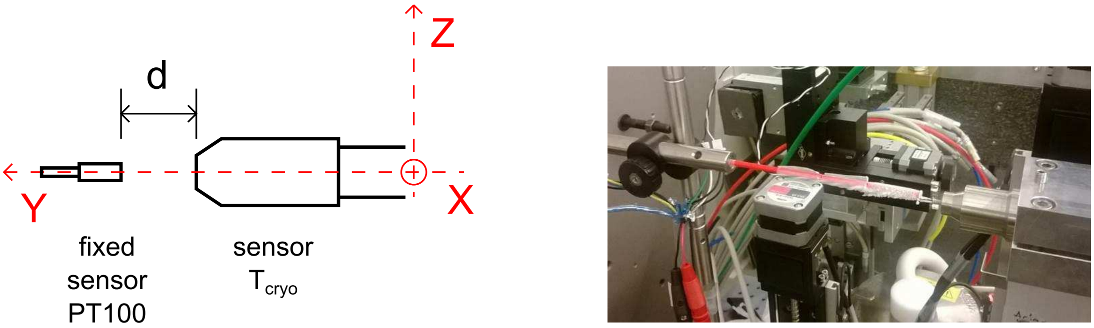

Figure 10: Sketch on the left side shows how the PT100 was positioned with respect to the cryocooler's nozzle. Right hand photo shows the actual situation at the beamline.

As for the first experiment the temperature of the cryocooler was set to T_{cryo}=90 K. Further we positioned the PT100 3 mm away from the nozzle (d = 3 mm). Two-dimensional (2D) scan was then performed within the $x - z$ plane. During the 2D scan the temperature was stabilized for 1 minute and 30 independent readings (every second) were averaged out thus yielding the mean value and its standard deviation.

One would expect that the temperature in the middle of the cold N_2 stream will be 90 K (see Fig.11). Surprisingly, the lowest temperature was around 110 K. It means that the difference between the temperature of the cold stream and the temperature detected by the sensor PT100 is around 20 K. The first idea was that there is some mistake with the temperature sensor PT100. However when dipping the PT100 sensor into liquid nitrogen bath it showed correct temperature of 77 K. After that the thermal contribution of the heater on the tip (prevents ice formation) of the nozzle was tested.

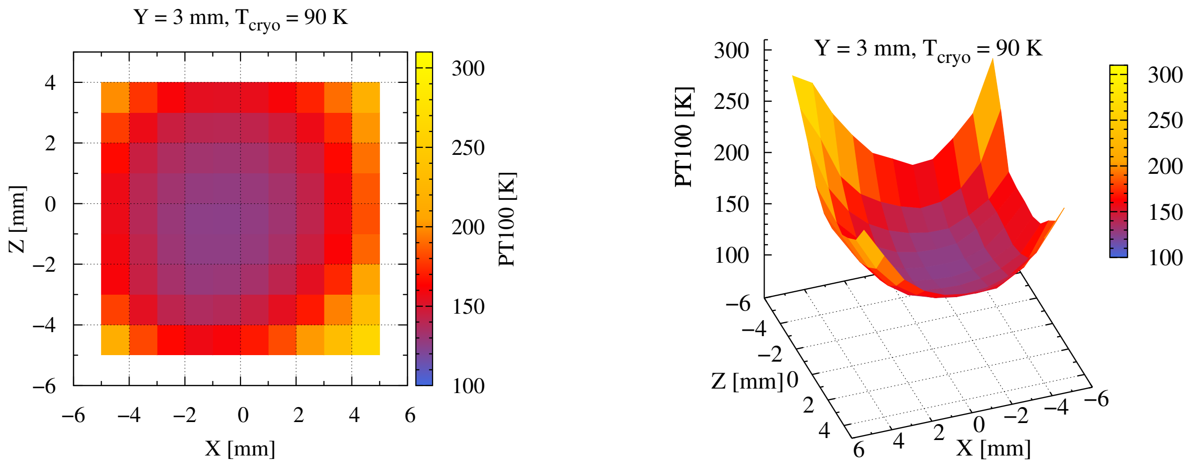

Figure 11: mapping the temperature gradient inthe $x - z$ plane perpendicular to the axis (y) of the cryocooler jet at T_{cryo}=90 K and $Y = 3$ mm.

This heater was switched off and similar measurement was repeated. However obtained results were same as in the first case. It seems that observed difference is due to another effect. It could be due to relatively small flow rate of N_2. Increasing the flow rate of the cold gas (increasing the voltage applied to the vaporizer inside the inner reservoir) could suppress the temperature difference.

In the next part the temperature gradients along the y axis were determined as a function of T_{cryo}. The temperature of the cryocooler was set to T_{cryo} and the distance d between the PT100 sensor and cryocooler's nozzle (see Fig.4) was varied. At every position there was stabilization time of 1 minute and 30 independent readings (every

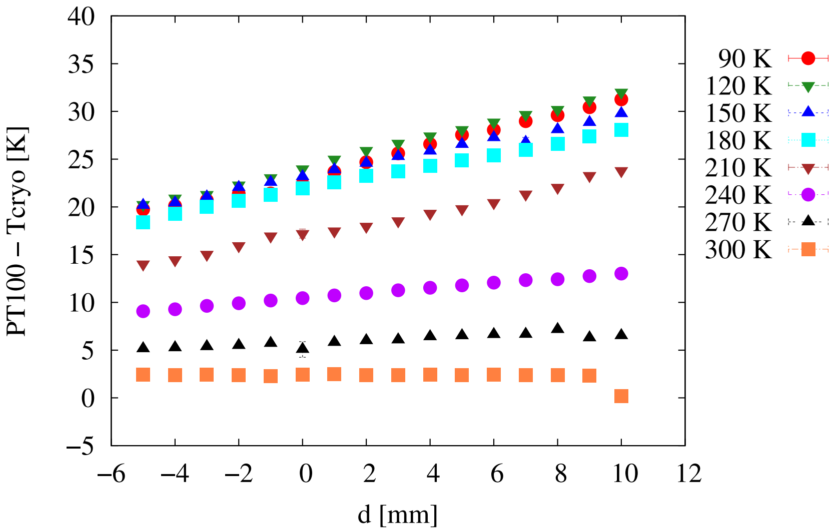

Figure 12: Evolution of the temperature difference between the PT100 and T_{cryo} with increasing distance for various temperatures of the cryocooler. Negative distance means that the PT100 was placed inside the nozzle.

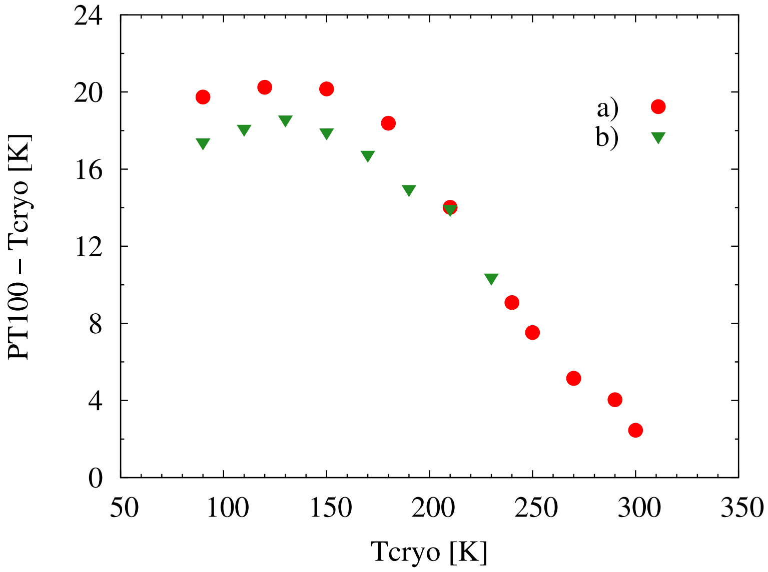

Figure 13: Comparison between values of temperature difference for d=-5 mm **a)** extracted from the plot shown in Fig.12 and **b)** from an independent measurement.

second) were averaged out thus yielding the mean value and its standard deviation. As one can see in Fig.12 the temperature difference is increasing with decreasing the temperature T_{cryo} of the cryocooler. Assuming that the sample is usually positioned 3 mm away from the nozzle, the temperature difference is not greater than 25 K.

To be sure the last measurement was done at distance $d = -5$ mm (the PT100 sensor was inside of the nozzle) for different temperatures T_{cryo}. As one can see from the Fig.13 the trend of dependence is similar and values are almost the same.

6 Conclusions

It was found that the Cryo-con cryocooler at the P02.1 beamline has consumption of liquid nitrogen of 0.84 cm/hour (at 22 V) and it depends on DC voltage applied to the N_2 vaporizer. Refined set of PID parameters allows smooth temperature change in the range between 90 and 300 K. Furthermore the temperature gradients around the cryocooler's nozzle were mapped (approximately 25 K difference at the lowest temperature). To suppress temperature difference on the sample it is suggested to increase the flow rate of cold N_2 stream, specially at lower temperatures, by increasing the voltage of the vaporizer. I hope obtained results will help future users to conduct successful in-situ XRD low temperature experiments at the P02.1 beamline.

Acknowledgement

Mainly, I would like to express big thanks to my supervisor Dr. Jozef Bednarčík for his time, knowledge and for his enthusiastic accession for science. He taught me a lot during these two months. I can say that he is as great teacher as a scientist. Moreover, I thank to Martin Domaracky, author of the Temperature control Python program [3], which I used for refining of the PID parameters and Jana Michaliková for her useful advices and help. Finally, my big thanks goes to all organizers of the DESY Summer Student Programme 2014, to all our lecturers and to all participants for such a great experience.

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[2] *PID theory explained*, `http://www.ni.com/white-paper/3782/en/`.

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