Eidgenössische Technische Hochschule Zürich
Swiss Federal Institute of Technology Zurich

Quantum Device Lab

Semester Thesis

Automatized Liquid Nitrogen Refilling of a Cryogenically-Cooled Supersonic Helium Source

by Ants Remm

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Abstract

The Rydberg experiment examines the interactions between helium atoms in Rydberg states and static or microwave fields emanating from a chip surface. The properties of the helium atoms in a supersonic beam, like temperature and speed, benefit in many ways from cooling down the atom beam source. To facilitate the cooling process an automatic liquid nitrogen refilling device including the necessary piping, sensors, control hardware and software was developed and integrated into the setup. The stability of the atom beam was analysed during refilling with the new system and no deviation from normal operation was found.

Contents

1 Introduction

As current computational devices approach the single atom limit, Moore's law does not hold any more [1]. A way to meet the ever-increasing need for computational power is to turn to alternative computation paradigms, such as quantum computation. This enables the use of new kinds of quantum algorithms which can in theory outperform classical algorithms in computational speed exponentially [2]. But there are still many obstacles that must be overcome before any real speedup can be achieved compared to a classical computer. DiVincenzo summarized the five main requirements for quantum computation [3]:

1) control over Hilbert space, for example in the form of well-defined extensible qubits
2) consistent state initialization
3) low decoherence times
4) a universal set of quantum gates
5) quantum state read-out

and the two additionl criteria [4]:

6) change between stationary and ,,flying" qubits
7) transmit qubit values over large distances

Analysing superconducting qubits by these criteria, there is hope that criteria 1 to 5 can be met in the near future exploiting error-correcting architectures required for fault tolerant calculation [5]. Criteria 6 and 7, however, are not satisfied as superconducting qubits are inherently stationary and the coherent transport of microwaves over large distances requires cooling the waveguides to millikelvin temperatures.

One possible solution for interfacing superconducting qubits with optical photons, currently the best method for long distance quantum information transport [6], is to use Rydberg atoms. Because of their large dipole moment, Rydberg atoms can couple to the electric part of the microwave field of a superconducting resonator on a chip if they are brought close enough to the surface, with coupling rates g up to 300 MHz [7]. Using this coupling the qubit state can be transferred from a superconducting qubit to a photon in a coplanar microwave resonator, and then to a Rydberg atom and to an optical photon.

1.1 The Rydberg experiment

The work done for this thesis is on an experiment studying the interactions between helium atoms in Rydberg states and DC and microwave electric fields [8, 9, 10]. The experimental setup [7] as seen in Figure 1 composes of two main parts: a helium beam source chamber and the main chamber.

A dense cold supersonic beam is generated in the source chamber which is discussed in more detail in the following section.

In the main chamber the atoms are excited to Rydberg states, interact with the test sample in a stack of 7 disc-shaped electrodes, and the signal is detected using a multi-channel plate (MCP from hereafter) detector. This stack is kept at 3 K by a two stage pulse tube cooling system. For excitation to Rydberg states a tunable pulsed dye laser operating in the UV range (313 nm) is irradiated onto the atoms between the first two electrodes, used to create a homogeneous electric field. In the interaction region microwave pulses from a horn antenna or a chip surface induce Rydberg-Rydberg transitions. Finally the Rydberg atoms reach electrodes 3 and 4 used for field ionization. The resulting electron beam is then magnified for imaging using electrodes 5 to 7 in an Einzel lens configuration. The electrons are detected using a MCP detector combined with a phosphorous screen and a CCD sensor. The MCP, which consists of a front and a back plate with the channels in a chevron configuration, can also be used in integrating mode to measure the signal time traces with an oscilloscope.

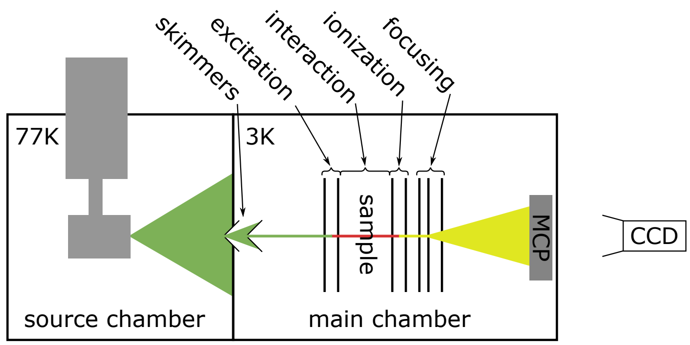


Figure 1: Sketch of the Rydberg experiment setup. Metastable helium atoms generated from the atom source (gray) are depicted in green, atoms in Rydberg states are in red, and the electrons are depicted yellow.

1.2 The helium source

The main component of the helium source is a pulsed valve operating at repetition rates of up to 1000 Hz. It releases pulses of helium atoms under high pressure (1.5–20 bar) into a vacuum chamber ($p \sim 10^{-7}$mbar). The valve is cooled to about 77 K with liquid nitrogen. Right after the release, the atoms undergo a supersonic expansion reducing their longitudinal temperature further to ∼500 mK. A dielectric barrier discharge excites part of the helium atoms to the metastable singlet $(1s)^1(2s)^1\ {}^1S_0$ and triplet $(1s)^1(2s)^1\ {}^3S_1$ states (named He* hereafter). Finally, ∼0.1 % of the atoms with the smallest transverse velocities pass through a set of two consecutively placed skimmers and head to the experimental part of the setup.

The liquid nitrogen used for cooling the valve is contained in a two part vessel. The smaller bottom reservoir made of stainless steel contains the valve and is connected by a vertical copper pipe to the bigger top reservoir, also made from stainless steel. This design reduces the effects of refilling on the valve performance.

Cooling the helium valve has several advantages. It reduces the speed of the atom pulses from $\sim 1800\,\mathrm{m\,s^{-1}}$ [7] to $\sim 900\,\mathrm{m\,s^{-1}}$ [11], and it reduces the beam temperature from ∼10 K [7] to about 0.4 K [11]. This reduces beam expansion in the longitudinal direction and, as a consequence, dephasing of atoms when interacting with microwave fields. Additionally, the slower beam allows for a longer interaction time of the sample and the Rydberg atoms.

2 The refilling system

Before this semester project was carried out, the helium source had to be manually refilled with liquid nitrogen approximately every two hours to keep it cold. This was done by pouring liquid nitrogen from a small dewar through a funnel into the source's reservoir. The need for constant attendance made it impossible to conduct long measurements.

To simplify and automate the operation of the experiment an automatic liquid nitrogen refilling system was developed. In this section its technical setup is described. As depicted in Figure 2, the main components of the refilling system are a 160 litre storage dewar of liquid nitrogen, piping to transport the nitrogen to the helium source, a cryogenic valve that controls the flow of nitrogen, and a control box that operates the cryogenic valve based on the inputs of two temperature sensors placed inside the source reservoir. The states of operation of the refilling system and the temperature of the helium source is monitored and logged by the experiment's logging computer.

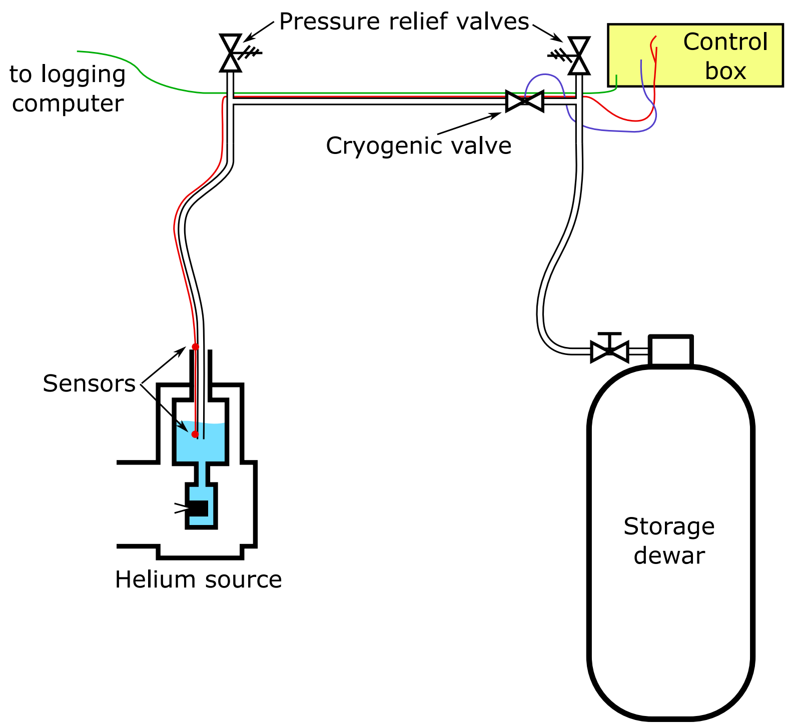


Figure 2: Schematic view of the refilling system, for details see text.

2.1 Liquid nitrogen transport

Liquid nitrogen is supplied to the experiment in the form of 160 litre dewars from the physics department shop that can be easily replaced when emptied. The liquid phase output of the dewar is connected to the source by flexible corrugated metal pipes and rigid quarter inch metal pipes. To thermally isolate the pipes from the ambient temperature the flexible and rigid parts are surrounded by Armaflex insulation tube and polystyrene foam respectively. All of the piping is covered in corrugated plastic tubing to protect it from mechanical damage.

The flow of nitrogen is controlled by a solenoid valve from ASCO, model number SCE263-205LT. It is operated by a 230 V AC input from the control box. The main sealing disc of the valve is made of polytetrafluoroethylene and it is verified for use with cryogenic liquids.

All the piping is attached to a Kanya profile frame surrounding the experimental setup. Due to the placement of the storage dewar, new Kanya profiles were added between the two existing frames of the optical tables in the laboratory.

2.2 The sensors

To measure the level of liquid nitrogen in the reservoir, two green light emitting diodes are used. The diodes are driven in the forward direction with a constant current of 10 mA and the voltage drop across them is measured. Immersing the diodes into liquid nitrogen from room temperature, the voltage drop rises from around 2.1 V to 6–9 V, depending on the specific diode at hand.

In the following paragraph I will present a short overview of the temperature dependence of a semiconductor diode's properties based on Xi *et al.* [12]. The Volt-Ampere characteristic of an ideal diode is given by the Shockley equation

$$I = I_S\left(e^{\frac{eV}{nk_\mathrm{B}T}} - 1\right) \tag{1}$$

with I being the current through the diode, V the voltage drop across the diode, T the junction temperature, I_S the saturation current, n a diode-specific dimensionless constant that varies around 1–2, k_B the Boltzmann constant and e the elementary charge. At first sight from this equation one might think that the voltage drop increases with temperature if the diode is driven with a constant current. This is not true however as the increase in saturation current I_S usually dominates over the decrease of the exponent in (1). The saturation current depends on the intrinsic carrier concentration, and the diffusion and the lifetime of electrons and holes, all of which depend strongly on the temperature.

The sensors are operated in binary mode. When the voltage drop across the diode is higher than a threshold value, the sensor is considered to be in liquid nitrogen and when it is below the threshold value, it is considered to be in a gaseous environment. The threshold values had to be found for each diode separately and were set to be 4.5 V and 7.1 V for the bottom and top sensor respectively.

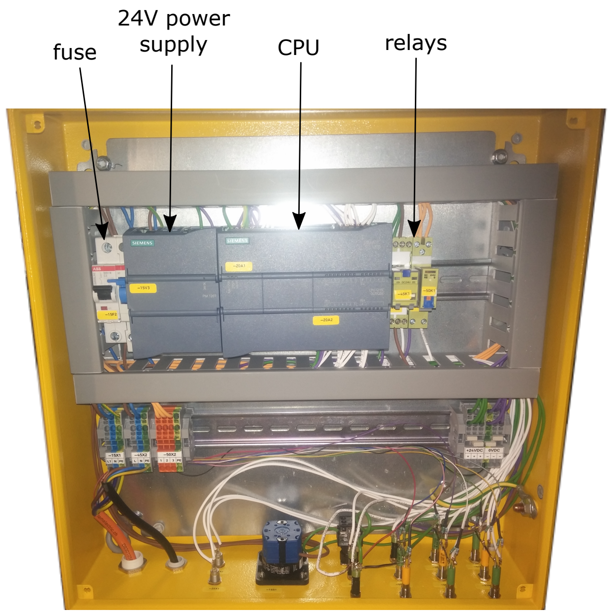
The bottom sensor, the position of which determines the minimum amount of liquid nitrogen in the reservoir during normal operation, is attached to the pipe that delivers liquid nitrogen to the reservoir. The top sensor is attached just above the reservoir, at the start of a tube that redirects overfilled nitrogen safely to a buffer dewar. This means that the top sensor becomes cold just before the reservoir starts overfilling. Before the final solution was found, three other ways of attaching the top sensor were tried out with the top sensor being inside the reservoir. These configurations were found problematic as during refilling the inflow of gas and liquid produces splatters of liquid nitrogen such that the top sensor inside the reservoir becomes cold before the actual liquid nitrogen level reaches it.

2.3 The control box

To operate the valve a custom control box was developed. It takes mains power and sensor voltages as inputs and outputs logging data and the valve control. The components and the front panel of the control box can be seen in Figure 3. It is based on a Siemens SIMATIC S7-1200 CPU 1215C programmable logic controller, designed for automation tasks. The CPU gets its power from a 24 V power supply, supplies a constant current to the sensors, reads the voltage drops, controls the relay that is connected to the valve, reads the front panel buttons, and shows the current state of the system on the front panel indicators. In addition to the relay used to drive the valve, there is an additional relay in the box, which can be used to operate an optional alarm signal. On the front panel there are indicators for the status of each sensor, the valve and the error state; a button to start the refilling process manually; a rotary switch for cutting the mains power; and a rotary switch for cutting 24 V power from the outputs of the CPU, that closes the valve but keeps the CPU running. Voltages for logging are read from the 24 V diodes using a $5.1\,\mathrm{k}\Omega$ and $18\,\mathrm{k}\Omega$ voltage divider that converts the voltages to an approximately 5 V range, suitable for reading with the National Instruments 9205 analog-digital converter attached to the logging PC.

The decice is programmed to open the valve when enough liquid nitrogen has evaporated in the reservoir that the bottom sensor becomes warm. When the top sensor becomes cold, the reservoir is full and the control box closes the valve, keeping the liquid nitrogen level between the two sensors. To program the CPU, Siemens' graphical programming environment "Totally Integrated Automation Portal" must be used. The full program code is saved in the documentation folder on the Rydberg drive, with a condensed version of the program being schematically shown in Figure 4. In short the program works as follows: The valve is opened when either enough liquid nitrogen has evaporated so that the bottom sensor becomes warm or when

(a)



(b)

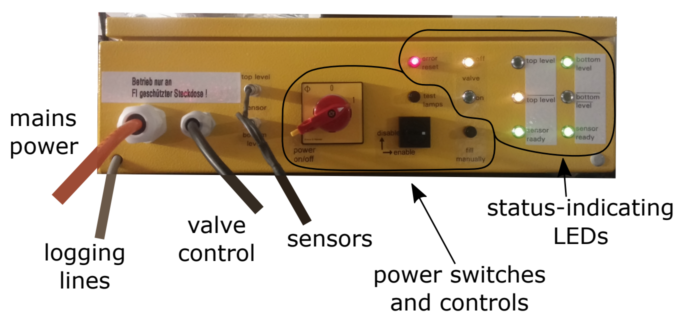


Figure 3: (a) The inside of the control box. (b) Front panel of the control box.

the manual refill button on the front panel is pressed. The valve closes when the top sensor becomes cold, which means that the liquid level has reached it, keeping the liquid level between the two sensors. Several safety measures were also implemented:

- If one of the sensors is disconnected or not working (voltage lower than 0.1 V or higher than 11.5 V) the valve is closed and an error is shown.
- If the valve is open longer than 250 s, the valve is closed and if the refilling was not started manually, an error is shown. This protects against situations where the liquid nitrogen is not directed to the reservoir or the storage dewar is empty.
- If the control box starts up, it requires the user to manually reset the error to begin operations. This prevents automatic refilling in case of an unexpected temporary power outage.
- To bring attention to a possible malfunction in the system, an error is shown at any time the bottom sensor is warm.

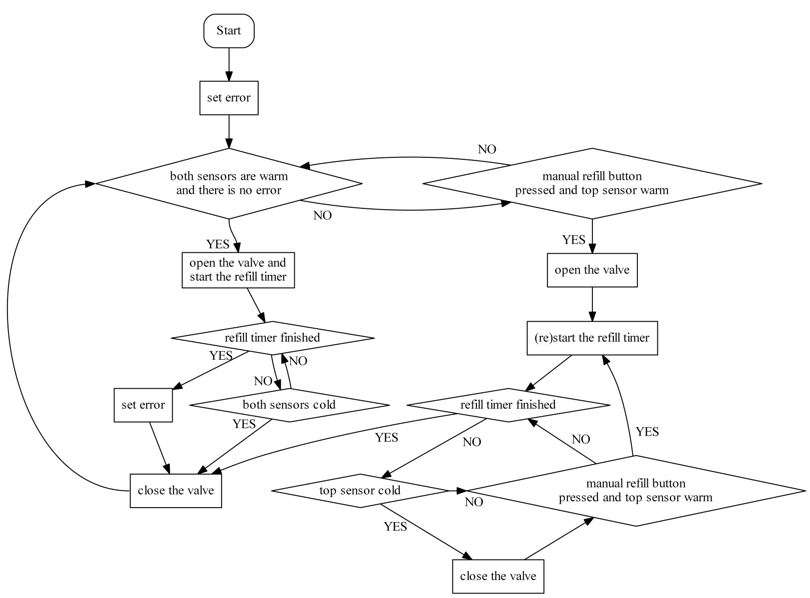


Figure 4: Flow chart of the program

2.4 Logging

To facilitate remote monitoring and subsequent tracking of potential problems with the source, a helium source monitoring and logging panel was added to the logging program. The logging program already had functionality to monitor and log different pressures and temperatures, the residual gas levels and the magnetic field in the experiment. The newly implemented panel shows the current state of the refilling system similarly to the control box front panel and the start and end times of recent refillings. It also shows the temperature of the helium source, measured with a K-type thermocouple. The thermocouple was previously connected to a multimeter that could measure the temperature down to only about 130 K. Now all the data is read from the National Instruments 9205 analog-digital converter. The voltage on the thermocouple is converted to temperature using Labview's built-in conversion table which was calibrated by liquid nitrogen (77 K), ice-water (273 K), room (294 K) and boiling water (373 K) temperatures.

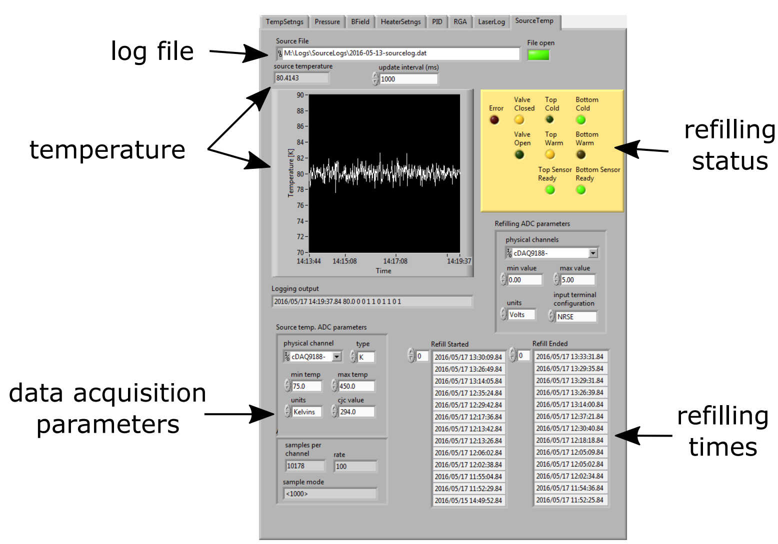


Figure 5: The panel added to the logging software.

3 Stability analysis

To see how the refilling process affects the properties of the atom beam and whether measurements could be carried out while refilling, I measured the properties of the helium beam of metastable He* states. A typical trace of the entire flight duration of the atom beam on the MCP can be seen in Figure 6.

For stability analysis traces were recorded over a duration of 53 minutes with the valve operating at 25 Hz. The results were saved approximately every 2.3 seconds after averaging over 25 atom pulses, giving 1406 timetraces in total. The liquid nitrogen valve was open for refilling between 19 min 27 s and 22 min 39 s from the beginning of the measurement. To better extract the beam properties, a narrower time window (vertical lines in Figure 6)

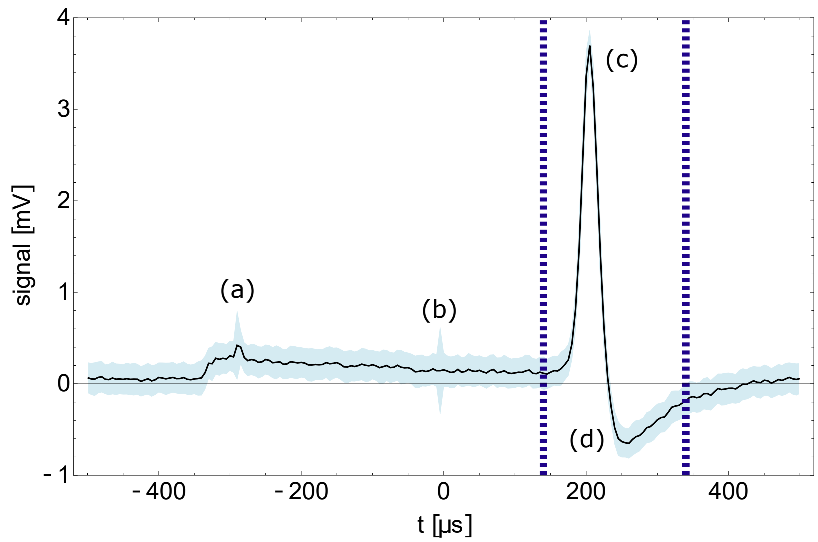


Figure 6: The shape of a typical timetrace measured by the MCP detector. The label (a) denotes a peak from the UV photons released during the dielectric barrier discharge. (b) indicates a spurious capacitive coupling of triggers to the high impedance line of the MCP used when measuring Rydberg states. (c) is the signal from the metastable atom pulse and (d) is a typical undershoot seen after the arrival of the atom pulse. The flight time of the atom pulse between peaks (a) and (c) is around 500 μs, which corresponds to velocity of about $900\,\mathrm{m\,s^{-1}}$, considering the 46 cm long flight distance. The dashed lines indicate a reduced time window for data acquisitions.

was set on the oscilloscope, giving a typical trace as in Figure 7a. Although the signal is quite noisy, it was confirmed that the noise is Gaussian and therefore the data could still be fitted with standard methods. The p value for the null hypothesis that the data is from a normal distribution is 0.9 according to the Cramr-von Mises test. The noise distribution can be seen in Figure 7b.

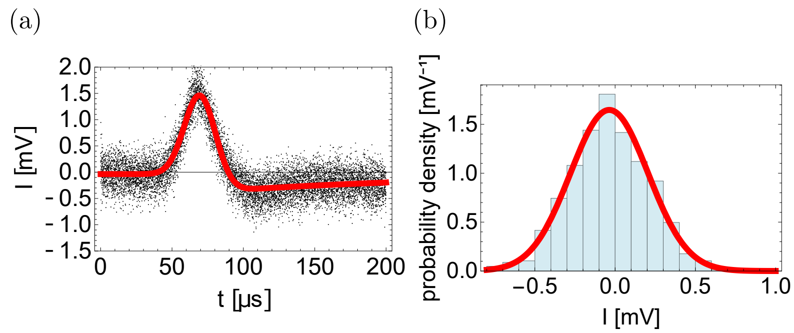


Figure 7: (a) A typical measured trace of the atom beam (in black) along with the fitted model (in red). (b) Histogram of noise distribution in the first 25 μs of a trace. A Gaussian fit is depicted in red.

All of the traces measured were fitted to the following model

$$\begin{aligned} I(t) &= e^{-\frac{(t-t_0)^2}{2\sigma^2}} * \left(S\delta(t) - \begin{cases} \frac{U}{\tau} e^{-\frac{t}{\tau}}, & \text{if } t > 0 \\ 0, & \text{otherwise} \end{cases} \right) + B \\ &= S e^{-\frac{(t-t_0)^2}{2\sigma^2}} - U \frac{\sigma}{\tau} \sqrt{\frac{\pi}{2}} e^{\frac{\sigma^2 - 2\tau(t-t_0)}{2\tau^2}} \operatorname{erfc}\left(\frac{\sigma^2 - \tau(t-t_0)}{\sqrt{2}\sigma\tau} \right) + B, \end{aligned} \tag{2}$$

where B, the background level, was taken to be the average of the signal during the first 25 μs. Here, S indicates the signal amplitude, t_0 the signal arrival time, σ the signal width, U the undershoot amplitude, and τ the undershoot time constant, which were fitted using the entire timetrace. The form of the fitting function and convolution kernel were chosen to model the effects of spatial charge build-up inside the MCP and saturation of the MCP's signal preamplifier [7].

Because not all the fits converged, the traces where the fitted signal amplitude S was negative or where the time constant was not in the reasonable range $0 < \tau < 10$ ms were dropped from further analysis, which amounted to about 17 % of the whole dataset. The distribution in time of removed traces has Poissonian noise (Figure 8a). It can be seen that the number of traces

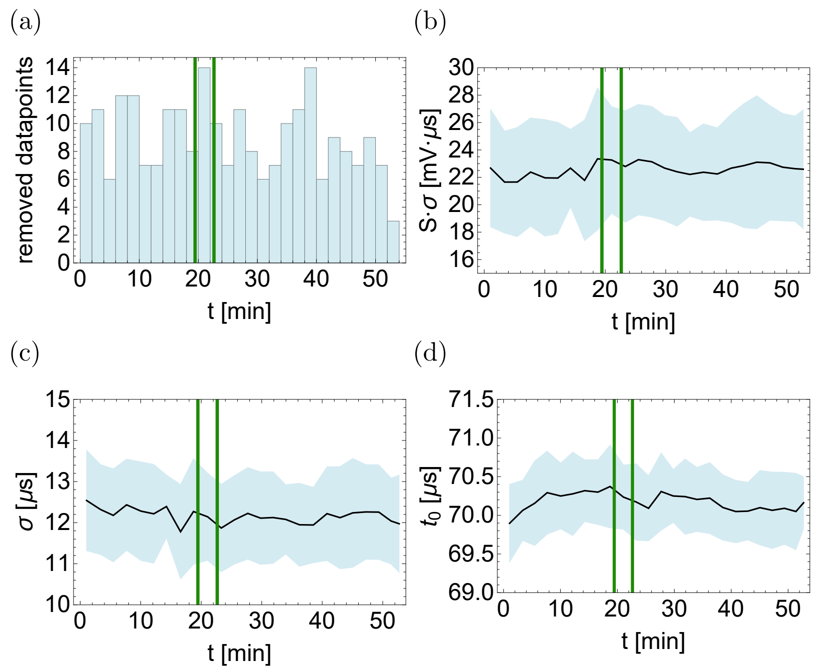


Figure 8: (a) The distribution of traces filtered out over time. (b)-(d) The time evolution of the optimal fit parameters for signal intensity, width and arrival time. The standard deviation of the best fit distribution is shown in solid around the mean. Green vertical lines indicate the time when the liquid nitrogen valve was open and the reservoir was being refilled.

filtered out during refilling (window indicated by green vertical lines) is a bit more than the average, but the deviation is within the natural variance of the distribution. The filtered timetraces were binned into bins of size 48 (129 s on average) and the mean and standard deviation of the fit parameters were extracted. The resulting plots of the signal area $S \cdot \sigma$, width σ and arrival time t_0 are depicted in figures 8b, 8c and 8d, respectively. Because the uncertainty of the fitting parameters from the fits are more than 4 times smaller than the variation within a bin for the three parameters $S \cdot \sigma$, σ and t_0, the fitting results can be taken to be exact. From the results as seen on Figure 8 we know that any change in the beam parameters during refilling is much smaller than the natural fluctuations and therefore we can safely assume that refilling does not affect the helium beam properties.

4 Conclusion and outlook

In the course of this semester project a system for automatically refilling liquid nitrogen for cooling the helium source of the Rydberg experiment was designed and developed. More specifically,

- a cryogenic valve and necessary piping were installed to the experiment
- different liquid nitrogen sensors were tested and mounted to the liquid nitrogen reservoir
- an electronic control box was assembled and programmed to operate the valve
- the refilling system as a whole was tested and set to working order
- monitoring and logging of the helium source temperature and refilling status was added to the experiment
- stability of the atom source was analysed

During the project I learned about the theory and motivation for the Rydberg experiment, gained a first experience with cryogenics and working in experimental physics environment. A more technical lesson was that the refilling process in the current setup is a wild process and it is hard to measure the nitrogen level during refilling. I also learned about the importance of planning steps with other people's schedules in mind.

Although the refilling system is now operational, there are still several improvements that can be considered. The program code for the control box, although functional, is currently not very clean as it is the result of several iterations of trying out different solutions. Another improvement is the placement of the top sensor, which is currently attached to the start of the overfilling redirection hose. Ideally there would be a small buffer vessel for liquid nitrogen, into which the sensor would be placed. This would make the top sensor more reliable. In longer term, additional liquid nitrogen and even helium refilling systems could be developed to replace manual refilling in other parts of the Quantum Device Lab.

Acknowledgements

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Appendix: Manual for the liquid nitrogen refilling system

Startup

- Make sure the sensors are positioned correctly with the bottom one (on a longer cable) attached to the liquid nitrogen output pipe and the top one (on the shorter cable) is at the start of the liquid nitrogen overflow redirection hose of the experiment.
- Make sure the output end of the piping is inside the reservoir of the helium source and the input is connected to a liquid nitrogen storage dewar's liquid phase output.
- Make sure that the sensors, the liquid nitrogen valve, mains power and optionally logging cables are connected to the control box.
- Open the valve on the storage dewar.
- Turn on the control box and press the reset error button. The refilling should start.
- After 250 s the reservoir is probably not yet full and the overfilling timer error is triggered. Reset the error by pressing the button when this happens until the reservoir is really full (indicated by top sensor cold LED or overflow of liquid nitrogen).
- The system is now set up and next time the bottom sensor becomes warm, the refilling starts automatically.

Operation

The refilling should mostly be automatic and require no intervention other than refilling the storage dewar when it gets empty. There are a few things to note however:

- Always make sure that the control box is switched off when removing the liquid nitrogen output pipe from the reservoir!
- The error LED will light up when the bottom sensor is warm. This is normal if you are just starting to cool down the source and should not be concerned about.

- If in any situation it is necessary to refill the reservoir before the bottom sensor has become warm (e.g. if you have to remove the storage dewar and want to make sure the reservoir is full before that) the manual refill button can be used. It should be noted that once the top sensor has become cold and the refilling is stopped, manual refilling can only be restarted after 250 s has passed from the last press of the manual refill button.
- To stop liquid nitrogen refilling for a short time, the enable-disable switch can be used, which cuts the power from digital outputs of the CPU, but leaves the program running for a faster reboot.

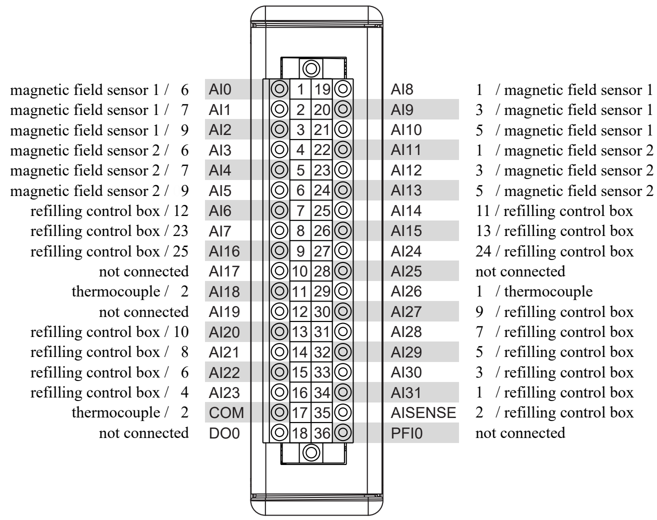
Logging

If there is ever need to rewire or debug something related to the NI 9205 digital-analog converter, then the current wiring is shown on the figure below.

The default parameters for the logging program (if they get accidentally changed for example) are as follows:

- Source temp. ADC parameters
  - physical channel - cDAQ9188-189E9A5Mod8: ai18
  - type - K
  - min temp - 75
  - max temp - 450
  - units - Kelvins
  - cjc value - 294
- Refilling ADC parameters
  - physical channels - cDAQ9188-189E9A5Mod8: ai20 – 23, 27 – 31
  - min value - 0
  - max value - 5
  - units - Volts
  - input terminal configuration - NRSE

(a)



(b)

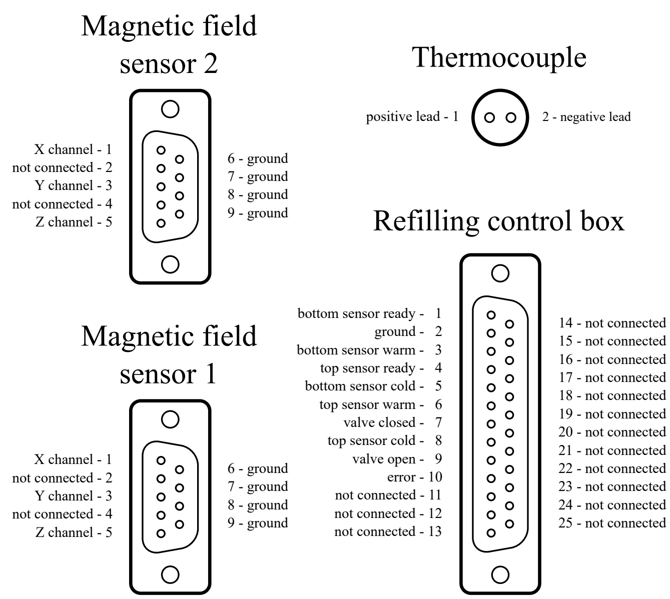


Figure: Wiring of logging cables on the NI 9205 analog-digital converter (a) and the front panel of the logging enclosure (b).

Troubleshooting

Here is a list of a few things that might be wrong with the refilling system and how to solve them.

- If you suspect some of the LEDs on the front panel of the control box are not working, you can test them with the test lamps button. In case the red LED was not working, it can be that it has just come out of its socket. Remove the button cap to check.

- If one of the ,,sensor ready" LEDs is not on and the corresponding ,,sensor cold" LED is blinking, it means that the corresponding sensor is either shorted or disconnected. Try repairing the connection or replacing the sensor. It is important to replace the diode sensor with one that has a voltage higher than the threshold voltage (4.1 V for bottom sensor, 7.1 V for top sensor) at 10 mA when dipped in liquid nitrogen. This can easily be tested with a voltmeter when the testable diode is connected to the control box as it acts as a 10 mA current source.

- If the system does not stop refilling even when the reservoir is overfilling (a few drops is OK), then the voltage when the diode is cold has dropped below the threshold for the top sensor and it has to be replaced (see the above comment on what to keep in mind).

- If the refilling cycle is too long (e.g. the atom beam properties are disturbed when the reservoir is almost empty) this means that the bottom sensor is placed too low on the liquid nitrogen output pipe. This can be mended by placing the bottom sensor higher up.

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