

# PROCEDURE

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Checked	
Approved	Chris Keith
Title	Operation of CLAS12 DNP Target for Run Group C in Hall B
Description	The procedure describes the operation and testing of a dynamically polarized target constructed for CLAS12 experiments comprising Run Group C inside Hall B.

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## 1. Revision History

Revision: 0	4/12/22	Original
Revision: 1	4/21/22	Modify section 6 for Hall B requirements; add wording for RadCon; corrections to target loading/unloading.

## 2. Definitions

- DAQ: Data AcQuisition
- DNP: Dynamic Nuclear Polarization
- EEL: Experimental Equipment Laboratory
- EIO: Extended Interaction Oscillator
- LHe: Liquid Helium
- NMR: Nuclear Magnetic Resonance
- ODH: Oxygen Deficiency Hazard
- OVC: Outer Vacuum Chamber/Can
- P&ID: Piping and Instrumentation Diagram
- PPE: Personal Protection Equipment
- RF: Radio Frequency

## 3. Purpose and Scope

The purpose of the procedure is to describe the processes required to:

- Prepare the DNP target system for cooling
- cool the system to 1 K
- load target samples into the system
- dynamically polarize the samples
- secure the system at the end of the operations

## 4. Facility

These process steps will occur in Hall B.

## 5. Authority

The authority to use this procedure will be determined by the JLab Target Group. Authority to alter this procedure is limited to the system owner.

## 6. Controls

### 6.1 Training and Briefing

The following are requirements to enter Hall B without an escort

- ES&H Orientation (SAF100)
- Rad Worker I Training or equivalent (must have been issued a dosimeter by JLab)
- ODH training (SAF103)
- Read and sign General Access Radiological Work Permit [RWP] (SAF801kd)
- Physics Division Work Governance (SAF116kd)
- Hall B Worker Safety Awareness Training (SAF111)

### 6.2 Personal Protection Equipment

The following PPE is required at all times in Hall B

- Hard hat
- Closed-toe shoes
- Long pants
- Personal dosimeter

### 6.3 Personal Protection Equipment

The following PPE is required whenever personnel are exposed to cryogenic hazards. Such hazards only exist when the 5 T solenoid magnet and 1 K refrigerator are initially cooled, and when target samples are loaded and removed from the 1 K refrigerator.

- Cryogenic gloves
- Cryogenic apron
- Face shield
- Safety glasses
- Long sleeves

## 7. Procedure Outline and Details

### 7.1 System Description

Operation of the CLAS12 polarized target system in Hall B will utilize the both the CLAS12 superconducting solenoid and Hall B LHe buffer dewar. Both are described by separated documents and not detailed herein. The polarized target comprised the following major subsystems:

1. A 1 K evaporation refrigerator and its control hardware and electronics;
2. A 6000 m<sup>3</sup>/hr pumping system for the refrigerator
3. A 140 GHz microwave system (comprising an EIO tube, power supply, and various waveguide components);

4. One or more NMR systems (comprising a Liverpool Q-meter, RF frequency generator, and DAQ electronics);
5. Polarizable target samples of frozen ammonia and non-polarized background samples of polyethylene and carbon (graphite). Tungsten wire samples will be used for calibrating the beam position and raster size;

## 7.2 Refrigerator Setup

The refrigerator system, whose major components are listed in section 7.1, shall be set up according to the P&ID shown below and verified by one or more of the System Experts listed in appendix A1. All piping connections shall be helium leak checked under the guidance of a System Expert. Using turbo pump MP8270, the refrigerator OVC shall be pumped to a vacuum level of  $1\text{e-}5$  torr or better.

Prior to cooling the refrigerator with liquid helium, it shall be prepared using the following Pump and Purge procedure.

1. Confirm that MV8260 is CLOSED and PI8260 reads 5 – 10 psig.
2. OPEN all other valves on the target's gas panel: MV8220, MV8230, MV8235, MV8240, MV8250.
3. OPEN solenoid valve SV8267A to send exhaust gas to the Hall B vent header (SV8267 should automatically CLOSE).
4. OPEN gate valve EV8210.
5. Turn ON gas panel pump MP8250 to start evacuating the system.
6. Turn ON the separator pump MP8230 and set flow controller FC8230 to 50 slpm.
7. Turn ON the refrigerator pumps in this order: MP8212A and MP8212B, then MP8211, and finally MP8210.
8. Pump until PI8250 indicates 20 mTorr or less, then CLOSE MV8250
9. Turn OFF the refrigerator pumps in the reverse order of step 7.
10. Turn OFF the separator pump.
11. OPEN MV860 and fill the system with helium gas to a pressure of 1 – 2 psig. Then CLOSE MV8260.
12. OPEN MV8250 and repeat steps 6, 7, 8, 9, 10, and 11.
13. Repeat step 12 two more times. Turn MP8250 OFF and close MV8235 and MV8240.
14. At this point, all pumps should be OFF. Valves MV8235, MV8240, and MV98250 should be CLOSED. All other gas panel valves should be OPEN, and gate valve EV8210 should be OPEN. The system is now ready for cooling with liquid helium.

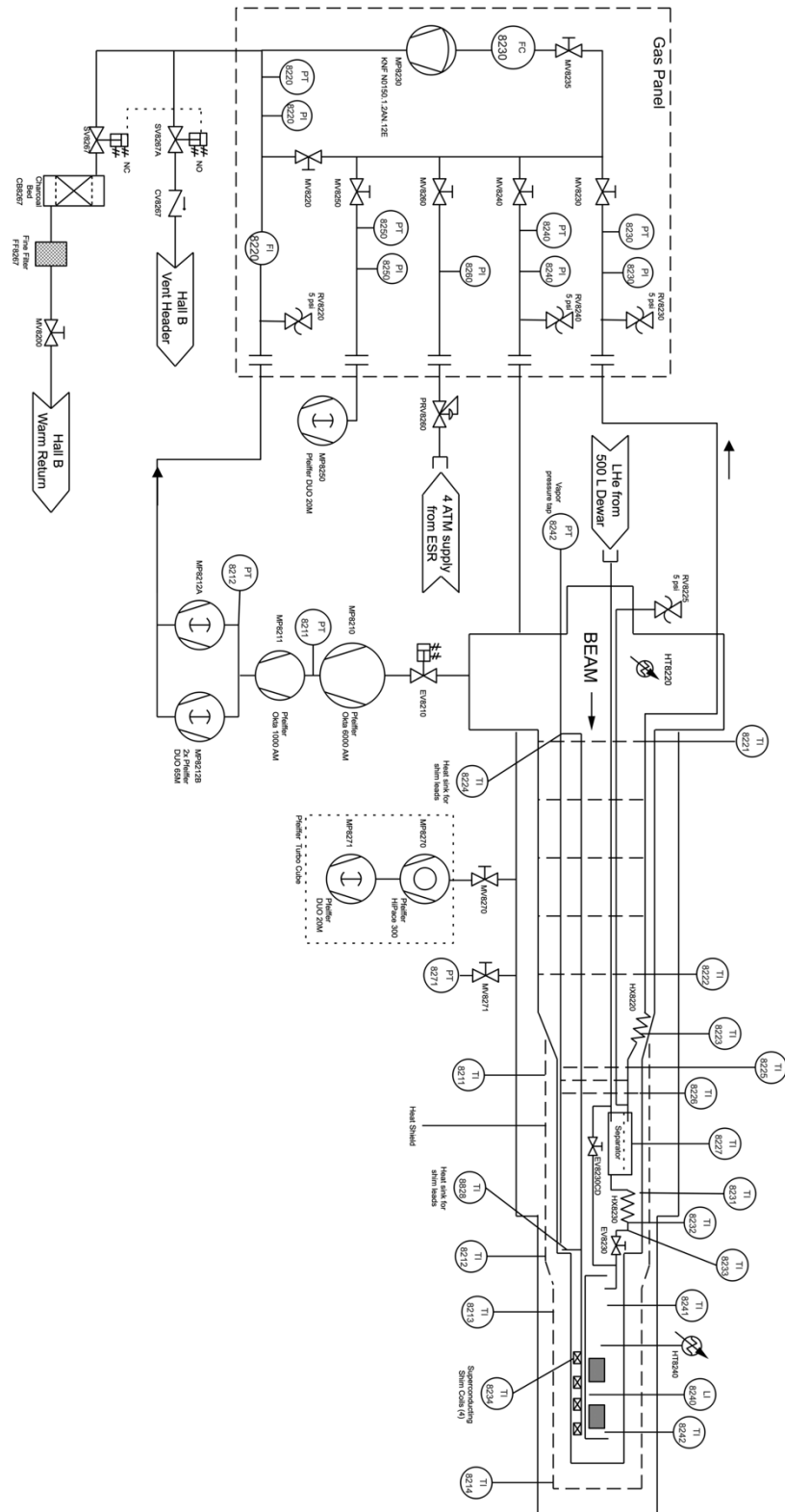


Figure 1: PI&D for the CLAS12 polarized target. Ignore references to the Hall B Vent Header and Warm Return.

### 7.3 Preparations for Liquid Helium Transfer

This portion of the procedure requires use of the dedicated, insulated transfer line for the 1K refrigerator. The line is specifically designed to operate with very low flow rates of liquid helium. This line has a  $\frac{3}{4}$ " OD, right-angle leg (withdrawal leg) for insertion into the Hall B LHe buffer dewar, a long flexible section, and a  $\frac{1}{2}$ " OD leg (delivery leg) for insertion into the 1 K refrigerator. The inner tube is only  $\frac{3}{16}$ " in diameter.

Follow the steps below to connect the LHe transfer line between the buffer dewar and the 1 K refrigerator. Standard cryogenic PPE is required: face shield, cryogenic gloves, and cryogenic apron. Two people and a six-foot step ladder are also required.

- 1) Confirm that PI8260 reads approximately 5 psig.
- 2) Open MV8230 and MV8260 to pressurize the separator to 5 psig. This can be confirmed using PI8230.
- 3) Using isopropyl alcohol and a clean cloth or rag, clean both legs of the transfer line.
- 4) Confirm that the insulating vacuum for the transfer line has been evacuated. If not, do so, pumping the line down to a vacuum  $1\text{e-}4$  torr or lower.
- 5) Locate the 6' ladder near the buffer dewar.
- 6) Set the operating pressure for the buffer dewar to 2 psig (1150 mbar) and wait for the pressure to equilibrate at this value. At this point you may open the 2 psig relief valve on the dewar. Gas may slowly escape through the relief valve, but this normal.
- 7) The first person should scale the ladder. The second person should hand the dewar-side leg of the transfer line to the first person.
- 8) The second person should remove the  $\frac{3}{4}$ " compression fitting plug at the top of the dewar. The first person should then insert the transfer line about 6" past the compression fitting at the top of the valve. Snug the compression fitting, but do not tighten it fully. DO NOT insert the line all the way to the bottom of the dewar yet.
- 9) The second person should confirm that warm helium gas is exiting ("purging") from the delivery leg of the transfer line, remove the  $\frac{1}{2}$ " compression fitting plug from the rear of the 1 K refrigerator, confirm warm gas is purging from this port, and fully insert the delivery leg into the refrigerator.
- 10) SLOWLY lower the transfer line down as far as possible. A reasonable rate is about 1 inch every 3 seconds. If the dewar's 2 psig pop-off valve opens vigorously, slow down. If it does not open, you can speed up slightly. After the transfer line has been inserted fully, the first person should tighten the compression fitting around the transfer line and dismount the ladder.

### 7.4 Starting the 1K refrigerator

After the system has been setup according to the previous sections, the 1 K refrigerator can be cooled.

1. OPEN both the run and bypass valve, EV8230 and EV8230CD, three turns;
2. Turn the separator pump MP8230 ON, and set the separator flow controller FC8230 to 40 slpm. This controls the siphon rate from the helium dewar;

3. Confirm that the helium gas pressure in the pressurization rig is 3-5 psig. CLOSE the isolation valve and OPEN gas inlet valve on the LHe dewar. OPEN the shut-off valve on the transfer line.
4. OPEN gate valve EV8210 and turn ON the refrigerator pumps in the following order: MP8212A and MP8212B, MP8211, and finally MP8210;
5. Routinely note all system temperatures and pressures as well as the refrigerator flow meter FI8220. As needed, adjust the regulator on the helium cylinder to maintain a pressure of 3-5 psi on the LHe dewar;
6. As the system cools, it will be necessary to slowly close bypass valve EV8230CD in order to maintain a refrigerator flow (FI8230) below 25 slpm;
7. When the target insert thermometers TI8242 and TI8241 cool below 4K, CLOSE bypass valve EV8230CD. Use run valve EV8230 to maintain the optimum system temperature and flow.

### 7.5 Loading Target Samples

**Prior to loading or unloading target samples, the beamline area around the target must be surveyed by a technician from the JLab Radiation Control Group (RadCon Tech) or their designee.** If necessary direct the RadCon tech to the target areas that will be accessed during the loading procedure. Do not proceed with the loading operation until the RadCon Tech gives their approval.

There are three parts to loading target samples into the refrigerator. First, the target bath is emptied and retracted to the loading position, and the fridge pumps are turned off. Second, the refrigerator is back-filled and purged with cold helium gas from the Run and Bypass valves, and target samples are exchanged. Third, the insert is put back into the refrigerator and the pumps restarted.

#### PART ONE:

1. Turn off the Fridge Flow Heater;
2. CLOSE both the Run and Bypass valves. Turn on the bath heater to 100% on the MEDIUM setting (about 1.3 W).
3. Wait for the bath temperatures to warm above 2 K and the refrigerator pressure to drop below  $1\text{e-}3$  torr. Turn OFF the bath heater.
4. Retract the target insert to the location of the load lock port on top of the refrigerator;
5. CLOSE gate valve EV8210 and turn OFF the refrigerator roots pumps: MP8210 and MP8211;

#### PART TWO:

6. Don the PPE listed in Section 6.3;
7. Place both the Run and Bypass valves in the Manual control mode and open them 5 turns. Bleed the refrigerator up to a pressure of approximately 1100 mbar. Relief valve RV8225 will vent helium and safely maintain this pressure;
8. OPEN the load lock port and remove the target sample into the insert using the long-handled loading tool. Place the sample into the temporary LN2 dewar. Close the load lock port as quickly as possible;



9. Extract the new sample from the LN2 dewar, OPEN the Load Lock port and place the sample in the bath. Close the Load Lock port as quickly as possible;

PART THREE:

10. OPEN gate valve EV8210 and insert the target bath to the In-beam position;
11. Put the Bypass valve in PID mode and control the fridge flow FI8220 at 60 slpm;
12. OPEN the Run valve to 5 turns;
13. Turn ON the refrigerator roots pumps in the following order: MP8211 followed by MP8210;
14. As the refrigerator cools, it may be necessary to lower the fridge flow setpoint from 60 slpm to 50, 40, 30 etc. When the fridge cools to 2 K, place the Bypass valve in Manual mode and close it to 0 turns.
15. Place the Run valve in PID mode to control the bath level LL8240 at 60%.
16. Turn ON the fridge flow heater.

After the refrigerator is cold ( $\sim 1$  K) and running in a stable manner, help the RadCon Tech perform a radiologic survey of the old target sample before it is placed in the long-term storage dewar.

## 7.6 NMR Set-up

Refer to the figure below for the standard NMR set-up. This should be confirmed by a Subject Matter Expert.

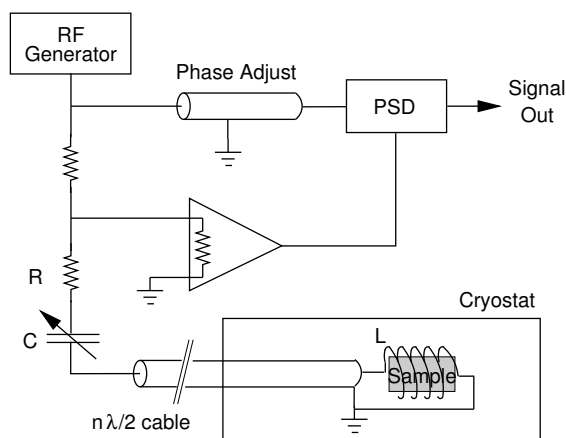


Figure 2: NMR setup for the DNP target.

### 7.7 Microwave Set-up

Refer to the figure below for the standard microwave set-up. This should be confirmed by a Subject Expert.

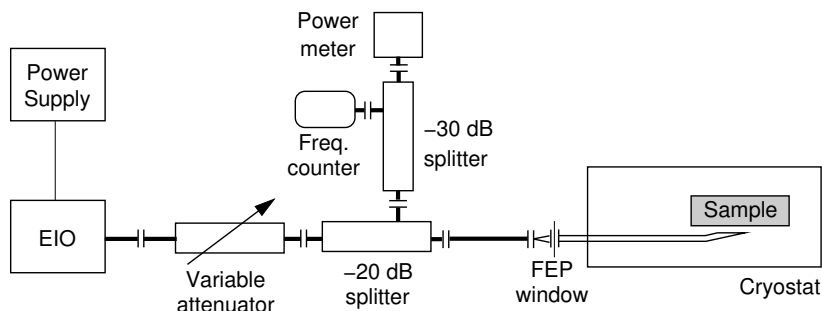


Figure 3: Microwave setup for DNP target.

### 7.8 Polarizing Target Samples

1. Request that the CLAS12 solenoid be energized to 5.00 T.
2. Turn ON the microwave source and adjust the frequency to the desired value. Approximate frequencies for positive (negative) polarization are 136.58 GHz (136.89 GHz).
3. Monitor the polarization using the NMR system, and adjust the microwave frequency to maximize the NMR signal size.

## 8. Emergency Procedures

In case of a life-threatening injury, personnel should immediately call 911 and contact the Jefferson Lab Guard Shack at x5822.

## A1. System Experts

The following is a list of System Experts for the CLAS12 Polarized Target. Only the system owner, Chris Keith, shall have the right to add or remove personnel from this list.

- Chris Keith
- David Meekins
- James Maxwell
- James Brock
- Chris Carlin
- Victoria Lagerquist
- Pushpa Pandey