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# Evaluation and redesign of a low pressure $LN_2$ Phase Separator

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**Abstract.** This study investigates operational instabilities in a low-pressure liquid nitrogen phase separator ($LN_2PS$) supplying a MAS-DNP NMR heat exchanger at the National High Magnetic Field Laboratory. The current system exhibits unstable behavior during filling cycles, requiring frequent manual valve adjustments to prevent cyclical overfilling and boil-off venting. A thermodynamic analysis was performed to characterize the phase behavior of the $LN_2PS$ during fill cycles. Approximately 8.4% of the input $LN_2$ undergoes flash vaporization via depressurization when entering the phase separator. Further analysis identified ice-ball formation on the primary back-pressure regulator as the dominant source of instability, where icing impedes valve closure and accelerates depressurization, causing cyclical and inefficient filling cycles. A redesigned configuration is presented in this paper that removes the low-pressure regulator positioned nearest the $LN_2$ inlet, relocates the remaining regulator downstream of the vapor buffer vessel, and incorporates a tee-split venting configuration. The goal of this revised configuration is to isolate the safety relief valve from the source of ice formation, separate steady-state pressure regulation from fill-cycle venting and ensure a stable $LN_2$ output stream to the MAS heat exchanger. The proposed configuration is expected to enhance operational stability, reduce boil-off inefficiencies, and mitigate safety risks associated with valve icing. The experimental evaluation of the modified $LN_2PS$ configuration will be reported in future studies.

## 1. Introduction

The NMR wing of the National High Magnetic Field Lab utilizes a piece of equipment enabling Magic-Angle Spinning Dynamic Nuclear Polarization, or MAS-DNP [1]. This specialty NMR technique uses an array of instrumentation to improve NMR resolution by 1 to 3 orders of magnitude. Sparing technical details on the analytical amplification method, the MAS-NMR operation parameters possess a strict set of requirements during sample runs.

One such requirement depends on a stable flowrate of low pressure (5 psi) liquid nitrogen fed into a cold bath heat exchanger for rapid cool-down of room temperature nitrogen gas to about 100 K. From the heat-exchanger, the nitrogen gas is split into three separate paths (bearing, drive and temperature) and used to spin and cool the sample at a set frequency during MAS-NMR analysis [2]. The strict parameters servicing the heat exchanger are to avoid condensation of the crossflow nitrogen gas (spin-gas); where, should the spin-gas phase shift to fluid, damage to the sample spin drive will result in catastrophic fashion.

The flow-regulated $LN_2$ supply is delivered via a liquid nitrogen phase separator ($LN_2PS$) located on the roof above the NMR Facility at the National High Magnetic Field Laboratory (MagLab). The $LN_2PS$ consists of a vacuum-jacketed, 20 L two-phase pot that functions as a fluid-regulated dewar, providing stable $LN_2$ stream to the MAS cold trap. Although the system design is conceptually simple, maintaining efficient and reliable operation has proven difficult. During repeated filling cycles, the $LN_2PS$ frequently exhibits instability in regulating its internal fluid volume. The system struggles to maintain fluid level and pressure within the vessel, which is observed as cyclical filling and venting of boil-off vapor within the $LN_2PS$. As illustrated in Figure 1, this behaviour frequently leads to ice-ball formation on the apparatus. Such ice buildup further exacerbates the problem by impeding valve function, accelerating depressurization and driving the system into a state of persistent instability.

**Figure 1.** Ice ball formation on the $LN_2$ phase separator (highlighted in dashed red box), leading to increased boil-off through pressure regulator valve impediment.

This study investigates the thermodynamic characteristics of the current phase separator and proposes a new configuration with the goal of improving the efficiency, performance and the safety of the $LN_2$ phase separator.

## 2. $LN_2PS$ System Design Schematic

The $LN_2PS$ delivers gravity-fed liquid nitrogen to the MAS heat exchanger, where its input $LN_2$ is supplied via a R50 VJ piping from a 6000-gallon bulk storage tank. $LN_2$ delivered directly from a high-pressure source is unsuitable for the MAS heat exchanger; therefore, the primary function of the $LN_2PS$ is to reduce the saturation pressure of $LN_2$ from the bulk storage tank (3.4 bara) to the lower pressure required at the point of use. The high-pressure $LN_2$ stream flows into the vessel and undergoes phase separation due to the volumetric expansion and saturation pressure being lowered, where the $LN_2PS$ maintains an internal pressure of about 1.3 bara. Vapor generated from nitrogen boil-off is vented, maintaining the set pressure of 5 psi for the consistent gravity-fed $LN_2$ delivery stream.

Liquid nitrogen fluid level within the $LN_2PS$ is controlled by a fluid level sensor actuating a diaphragmed VJ globe valve with a pneumatic actuator. The system pressure is regulated through dual back-pressure regulators set at 5 psi and 7 psi, with a vent solenoid valve set at 14 psi to minimize overpressure from flash vaporization during fill cycles. It has a primary fluid tank capacity of 20 L, with an additional 30 L overhead gas volume buffer tank, and maintains a fluid level of 80 %, or roughly 16 L; where heuristics begin filling at 60 %, adding about 4 L of $LN_2$ per fill. Figure 2 below outlines the phase separator design and highlights major components of the system.

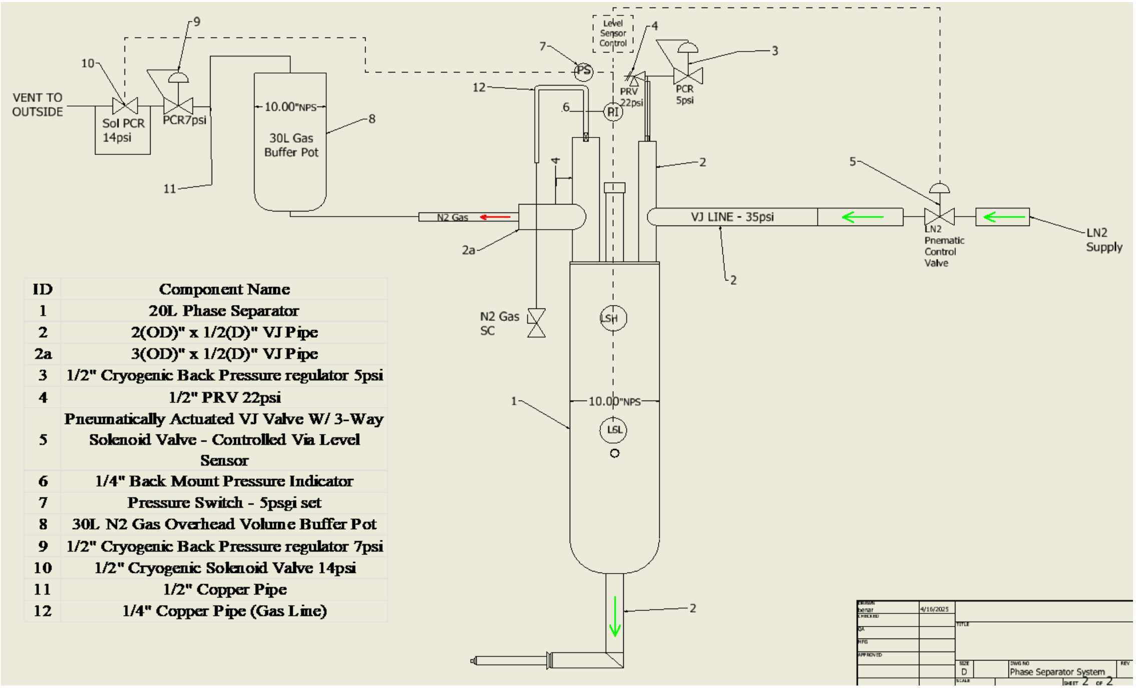


**Figure 2.** $LN_2$PS Schematic [3]: Current configuration leads to ice-ball formation on component ID '3'.

## 3. Thermodynamic State Analysis

This chapter presents a T–v diagram with the purpose of characterizing the specific volume envelope of the phase separator during fill cycles in relation to the fluid–vapor saturation boundary. Under the assumption that the system operates isothermally with a vented configuration (constant temperature and pressure), the diagram provides a thermodynamic basis for visualizing phase behaviour and quantifying mixture volume changes throughout the fill process. The saturation curve data employed for diagram construction are presented in Table 1 below.

**Table 1.** Saturated LN2 fluid properties for flash vaporization [3]

| LN2 | Pressure (kPa) | Enthalpy (kJ/kg) | $T_{Sat}$ (K) |
|---|---|---|---|
| Upstream fluid | 341.3 | -96.9 | 89.4 |
| Upstream fluid at Fill Valve (Pascals Law [a]) | 295.5 (Static Pressure) | -100.4 | 87.7 |
| Phase Separator (fluid) | 135.8 | -116.9 | 79.9 |

[a] Due to the elevation increase of the phase separator in reference to the $LN_2$ bulk storage tank, Pascals Law was utilized to determine the static pressure head at the fill actuator valve.

*3.1 Flash Vaporization Fraction Potential*

Flash vaporization, or adiabatic flash, is the partial vapor that occurs when a saturated liquid (specifically the saturated $LN_2$ feeding the phase separator), undergoes a sharp reduction in pressure when entering the phase separator by going from 295 kPa to 136 kPa. The fraction of supplied $LN_2$ fluid mass which flash vaporizes is an energy balance ratio of the difference in enthalpy of the fluid upstream and downstream, divided by the enthalpy difference of the downstream vapor and downstream liquid (essentially the latent heat of vaporization). The flash vaporization potential was calculated by Equation 1 below:

$$\chi_{flash} = \frac{h_u^f - h_d^f}{h_d^v - h_d^f} = \frac{-96.9[\frac{kJ}{kg}] - \left(-116.9[\frac{kJ}{kg}]\right)}{79.1[\frac{kJ}{kg}] - \left(-116.9[\frac{kJ}{kg}]\right)} = 0.084 \quad (1)$$

where enthalpy of the upstream fluid $h_u^f$ is estimated to be 96.9 kJ/kg, enthalpy of the downstream fluid $h_d^f$ is estimated to be 116.9 kJ/kg and the enthalpy of the downstream vapor $h_d^v$ is estimated to be 79.1 kJ/kg. Flash vaporization potential was calculated to be 0.084, meaning 8.4 % of the fluid mass entering the phase separator immediately flashes to a vapor. This is a typical value for most phase separators; however, it may have implications on the phase state equilibrium during cyclical filling cycles, especially during failed open valve scenarios due to increased boil-off from rapid depressurization.

*3.2 Evaluating Thermodynamic State During Pressure Regulation Failure*

In addition to flash vaporization, we wanted to visualize the specific volume changes resulting from $LN_2$PS pressure deregulation from the ice-ball formation apparent in Figure 1. This ice ball causes the primary back pressure regulating valve to become impinged and freeze open. This causes the system to rapidly depressurize in between fill cycles and is the dominant culprit in system instability.

Internally, this 'valve stuck open' scenario causes the pressure within the vessel to quickly drop from 14 psi to 5 psi over the course of about 3 seconds, leading to rapid fluid boiloff. This is indicated on the T-v diagram in Figure 3 and was calculated synonymously to flash vaporization. , the rapid depressurization is enthalpically identical to the initial flash vaporization, simply multiplying our mass boiloff during fill cycles by a factor of 2.

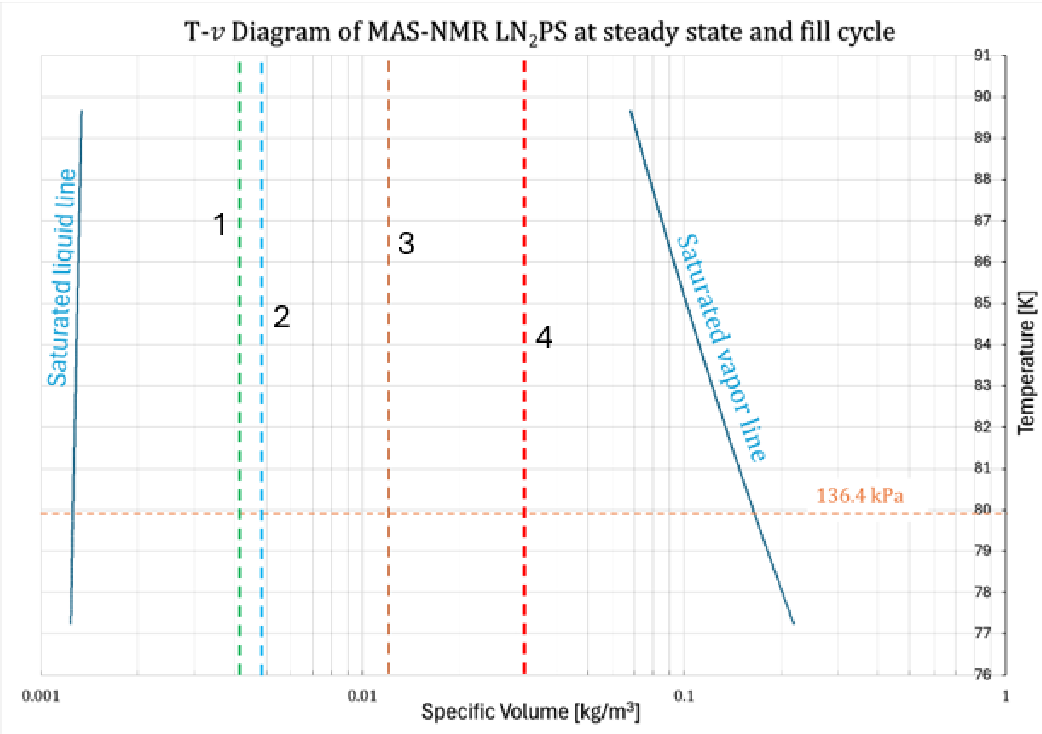


**Figure 3.** T-v Diagram MAS-NMR $LN_2PS$ at steady state and after fill cycle with: constant specific volume line at 80 % fluid volume (green dashed-line 1), constant specific volume line at 60 % fluid volume (blue dashed-line 2), constant specific volume line at 60 % fluid volume with fill cycle and flash vaporization (brown dashed-line 3), and constant specific volume line at 60 % fluid volume with fill cycle, flash vaporization and rapid depressurization (red dashed-line 4).

The T-v Diagram outlines the thermodynamic state during various stages of filling cycles. When not actively filling, the $LN_2PS$ maintains an internal fluid volume between 60 and 80 percent tank capacity; indicated by the blue and green dotted lines, respectively. Immediately after a fill cycle, the flash vaporization point is indicated with a brown dotted line. Accounting for the rapid depressurization with iced over valve impingement, the thermodynamic state of the vessel moves further to the vapor saturation curve and is indicated by a red dotted line. At this point, the vapor quality of the phase separator system is nearly 20 %.

## 4. Proposed $LN_2$ Phase Separator Reconfiguration

The fundamental objective in redesigning the $LN_2PS$ is to eliminate ice-ball formation points which impact valve function and system stability. In the current configuration, the 5-psi back pressure regulator (BPR) shown in Figure 4, located nearest the $LN_2$ supply stream, is prone to icing under normal operating conditions. Depressurization reduces the $LN_2PS$ pressure from 14 psi to 5 psi within approximately 3 sec. This rapid pressure drop induces significant nitrogen boil-off as the system absorbs latent heat to maintain isenthalpic equilibrium, resulting in the localized ice formation. This ice-ball presents two primary issues: Compromising system pressure regulation when the ice-ball impedes the BPRs ability to close fully and the safety concern arising from the ice-ball's proximity to the safety relief valve.

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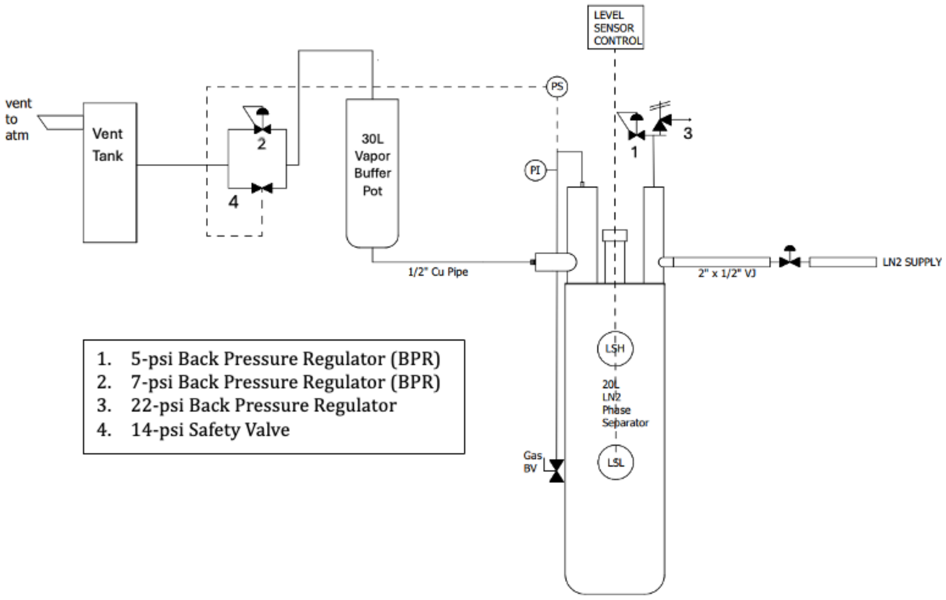


**Figure 4.** LN2PS current configuration: operation becomes chaotic once BPR ices over, leading pressure regulation failure. This depressurization brings the LN2PS from 14 psi to 5 psi in about 3 seconds, leading to high boiloff to maintain isenthalpic balance. The primary back pressure regulators and safety valve are specified by the number in the figure above.

These two concerns are alleviated in the proposed reconfiguration presented in Figure 5, which involves the removal of the 5-psi BPR, and redesign of the downstream venting framework with installation of a tee splitting point. This tee split creates two distinct exhaust regions, one for venting during fill cycles, another for maintaining pressure regulation during steady state boil-off. In Figure 4, depicting the current $LN_2PS$ schematic, the 14-psi safety valve and the 7-psi back pressure regulator are installed in series between the 30 L vapor buffer pot and atmospheric vent tank. Figure 5 depicts the reconfiguration by moving the 14-psi safety valve downstream from the ½” FNTP TEE, which serves to assist in pressure regulation during flash vaporization from fill cycles. Meanwhile, the 7-psi BPR, is moved downstream from the 30L vapor buffer pot, and serves pressure regulation during steady state boiloff.

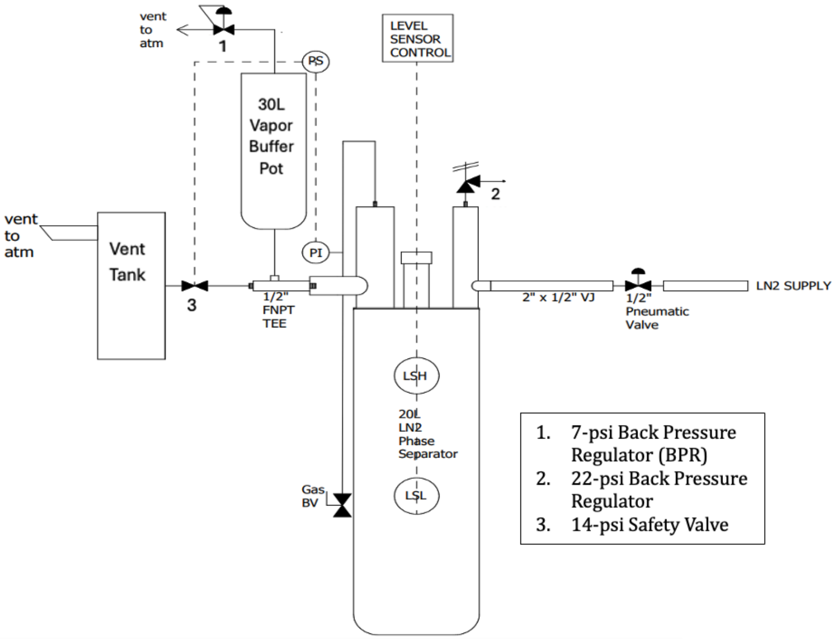


**Figure 5.** Proposed configuration to eliminate potential for ice over of back pressure regulator. The primary back pressure regulators and safety valve are specified by the number in the figure above.

## 5. Conclusion

The proposed changes to the $LN_2PS$ have not yet been implemented, but the new setup is expected to enhance the operational performance while improving the safety and efficiency of the system. First, removal of the back pressure regulator adjacent to the 22-psi safety relief valve will isolate the PRV, mitigating the risk of ice-induced valve freezing. The addition of a tee-fitting on the gas outlet, with the 14-psi solenoid valve vented to the primary vent tank, is expected to contain any liquid ejection during fill cycles. Finally, relocating the BPR downstream of the 30 L gas buffer vessel and split the vapor buffer pot from the vent tank should stabilize system pressure during steady-state boil-off, addressing a primary source of operational instability. The future study will be shared to present the experimental results of the modified $LN_2PS$ configuration.

## Acknowledgments

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