1240 (2022) 012118

# **Operational experience with the Proto-DUNE NP02 and NP04 large volume liquid argon cryostats and their cryogenic systems at CERN**

# J.Bremer<sup>1</sup>, M.Chalifour<sup>1</sup>, J.Creus-Prats<sup>2</sup>, C.Fabre<sup>1</sup>, D.Montanari<sup>2</sup>, M.Pezzetti<sup>1</sup>, F.Resnati<sup>1</sup> and M.Nessi<sup>1</sup>

<sup>1</sup> CERN, CH-1211 Geneva 23, Switzerland

<sup>2</sup> Fermi National Accelerator Laboratory, Batavia, IL 60510 United States

E-mail: johan.bremer@cern.ch

**Abstract.** The far Detector of the Deep Underground Neutrino Experiment (DUNE) will be housed in several large volume (about 12.500 m<sup>3</sup>) liquid argon cryostats. The design principle of these large cryostats, and of the cryogenic system belonging to them, are investigated through the design, construction and operation of a series of prototype installations.

The Neutrino Platform 02 (NP02) and 04 (NP04) cryostats, placed at CERN, contain DUNE proto-type detectors, each of them housed in an about 600 m<sup>3</sup> liquid argon bath. These cryostats, based on the membrane cryostat principle, and their cryogenic systems have been designed according to the DUNE principle. Measurements performed in these test stands shall confirm the foreseen heat loads into the cryostat systems entering via its walls, via detector cabling and via the cold electronics, shall confirm the low temperature gradient over the active detector volume and shall certify the liquid argon purification principle.

This paper introduces the requirements for the NP02 and NP04 cryostats and their cryogenic systems, describes the design principle applied to these two systems and gives an overview of the different modes in which the two systems have been operating. The experimental results are presented and discussed, and "lessons learned" for future installations are dawn.

#### 1. Introduction

CERN is a member of the international collaboration working on the DUNE experiment [1,2]. For this experiment it is foreseen that neutrinos are created by shooting a high intensity / high energy proton beam at a graphite target at Fermilab. The particles created by the interaction (pions) will be directed in a defined direction before they decay naturally in muons and neutrinos; The muons will be removed from this beam, while the neutrinos will continue to travel to the DUNE experiment situated at 1300 km from Fermilab in caverns located at about 1500 m underneath the surface. The neutrinos will be detected in liquid argon-based detectors placed in these underground caverns.

The detectors will be placed in large volume liquid argon cryostats (about 12500 m<sup>3</sup> per cryostat). Based on safety and operational requirements, a choice of a non-standard cryostat design has been made: the membrane cryostat [3]. In more conventional cryostats, the thermal insulation is guaranteed by a vacuum space between the vacuum vessel at room temperature and the cold vessel containing the cryogenic

Content from this work may be used under the terms of the Creative Commons Attribution 3.0 licence. Any further distribution of this work must maintain attribution to the author(s) and the title of the work, journal citation and DOI. Published under licence by IOP Publishing Ltd 1

liquid. In the design foreseen to be implemented at DUNE, the liquid argon is in direct contact with the stainless steel primary membrane (1.2 mm thickness) while the insulation is formed by layers of polyurethane foam insulation (see fig 1). Although this insulation method will result in a higher heat-load in normal operation, it excludes the risk of a vacuum failure, which can happen in the conventional cryostat design, leading to extreme heat load to the cryogenic liquid, and thus to a high evaporation rate. To diminish the risk of an eventual argon spill from the cold cryostat vessel to the warm out shell, a secondary membrane is present within the insulation material layer.





The NP02 and NP04 cryostats at CERN have been constructed to prototype the future DUNE cryostats and their cryogenic system [4]. During two cold runs lasting several years, the cryogenic performance of the cryostats has been tested, as well as the temperature stability and temperature gradient of the liquid argon baths housed in the cryostats. It has also be proven that the liquid argon purity level needed for the correct functioning of the detector could be reached with the help of the purity system as foreseen for the DUNE installations.

The internal cold vessel has the dimensions of  $(8 \times 8 \times 8) \text{ m}^3$ , while the outside warm metallic support structure, giving the necessary support to the inner stainless steel membrane through the insulation material has a dimension of  $(10 \times 10 \times 10) \text{ m}^3$ .

## 2. Demands for the cryostats and cryogenic system

The NP02 and NP04 cryostats have been defined based on the DUNE cryostat design. According to this design, the heat load entering the cryostat structure shall be smaller than 7.5 W/m<sup>2</sup>, while the heat load caused by the cabling entering the cryostat volume from the top, will of course dependent on the number of wires entering the liquid argon bath. The steel structure supporting the liquid argon through the membrane and the insulation material has been designed to withstand a maximum relative argon gas pressure of 350 mbar on top of a filled cryostat, while the membrane itself cannot withstand any pressures in the cold vessel lower than -35 mbar relative to the ambient pressure. For these reasons a safety valve has been installed on the cryostat argon volume, which releases the argon cryostat pressure to air in case the relative pressure rises above 350 mbar relative. The cryogenic system [5], regulating

CEC 2021		IOP Publishing
IOP Conf. Series: Materials Science and Engineering	1240 (2022) 012118	doi:10.1088/1757-899X/1240/1/012118

the cryostat gas pressure has thus also to be defined to be operational within these two extreme limits. To guarantee a stable detector signal independent from time and independent form the placement of the detector within the sensitive liquid argon volume, the temperature stability in the liquid argon bath over time needs to be lower than 100 mK, while the temperature gradient between any two pints within the sensitive volume shall be smaller than 0.5 K.

The operational status of the detectors housed in the cryostats is heavily depending on the free electron lifetime of the liquid argon within the cryostat, and thus directly on the electro negative impurity level of this liquid argon. The argon delivered to the test area has relatively high impurity levels ( $O_2 < 2ppm$ ,  $H_2O < 1 ppm$ ;  $N_2 < 2 ppm$ ), while the argon within the sensitive cryostat volume shall have an oxygen equivalent impurity level in the order of several 100's ppt. It is foreseen that the impurity level within the cryostat volume shall be lowered and maintained with the help of an active copper based purifier and a molecular sieve placed outside of the cryostat volume. The liquid argon is passed through this purifier system with the help of a liquid argon pump also placed outside the cryostat volume.

#### 3. Cryogenic system

The principle of the cryogenic system designed for the NP02 and for the NP04 prototype detectors is presented in Fig. 2.



Figure 2: Presentation of the operational principle of the NP02 and NP04 ProtoDUNE cryogenic systems

The pressure regulation of the argon bath volume will relieve argon gas, created by the overall heat load into the argon volume, through the gas lines connected to the signal chimneys (relatively small gas flow, sweeping through the room temperature signal wires volume, removing the oxygen degassing from the warm surfaces) and through a line directly connected to the cryostat gas volume (major cold argon gas flow). The small room temperature gas flow is passed through an active copper based purification bed, in which the oxygen content in the gas is reduced. Before passing this bed, the argon gas is also lead

through a molecular sieve to lower the water content present in this gas flow. After the purification cycle the chimney gas flow is, together with the major cold gaseous argon flow coming from the cryostat volume, lead to the liquid nitrogen based condenser. The liquid nitrogen pressure in this argon condenser is regulated such that the condensing argon is cooled to a temperature of about 87.3 K.

To assure that all the argon coming from the cryostat is passing through a cold purifier system, the liquid argon from the nitrogen condenser system is led to the inlet of a liquid argon pump based at the bottom of the cryostat, where it is mixed with liquid argon coming directly from the cryostat volume via the bottom cryostat connection. This liquid argon mix is then passed through a cold purifier system, based on the same principle as the above mentioned warm purifier. The liquid coming from this purifier system is passed through an argon phase separator after which the liquid argon flow is returned to the bottom of the cryostat, while the gaseous argon coming from this separator is fed to the already mentioned condenser system. The pressure in this phase separator is regulated such that the temperature of the liquid argon returned to the bottom of the cryostat is slightly warmer then the liquid already present in the cryostat. This temperature difference (and thus density difference) will lead to a smooth convection flow within the cryostat volume, leading to a small temperature gradient over the cryostat volume.

Both the warm and the cold purification systems can be re-activated by reducing their water / oxygen content. The molecular sieve can be regenerated by sending a warm argon gas flow in counter sense through the system which leads to the vaporization of the water content adsorbed by the molecular sieve, while the copper based purifier has to be flushed by an argon based warm gas flow, containing a hydrogen percentage of maximum 2%. The hydrogen will react with the formed copper oxide, creating a water vapor content and an active copper surface.

#### 4 Cool-down and normal operation procedures

Before the cool-down of a proto-DUNE cryostat can start, the air present in the cryostat volume has to be removed. Gaseous argon coming from the outside storage tank is entered at the bottom of the cryostat. The gas flow shall be regulated such that the vertical gas speed in the cryostat is around 1 m/s. At this speed the movement of the gas doesn't create any turbulences, while the speed is high enough to overcome any back diffusion. After about 12 renewals of the cryostat volume the oxygen content in the argon volume has dropped to below 5 ppm (see fig 3). From now onwards, the cryostat volume is circulated through the warm purifier, lowering the oxygen and water content in the cryostat's gas volume even further. From figure 3 it can also be seen that the nitrogen content in the cryostat's gas the nitrogen atoms. Once the cryostat oxygen content fell below 0.1 ppm is was decided to start with the cool-down of the cryostat.

During the cool-down cycle, when the maximum temperature difference between any two points within the detector volume shall be smaller than 50 K, gaseous and liquid argon flows are entered into a sprayer system placed at the top of the cryostat, creating a cold argon mist over the detector volume. At the same time a small flow of liquid argon is entered at the bottom of the cryostat, evaporating when coming in contact with the cryostat bottom, assuring an argon gas movement over the complete argon volume. Within a delay of less than four days the warmest point within the sensitive volume has been lowered to about 130 K liberating the system for the start of the liquid argon filling.

During this filling period a daily argon quantity of about 20 to 40 tons is delivered to the outside storage tank before being transferred into the cryostat. The oxygen content of the argon being delivered to the outside storage tank is checked before every transfer to assure that only argon with an oxygen content below 2 ppm is entered in the proto-DUNE cryogenic system. The pressure in the cryostat is during the transfer of the liquid argon into this volume regulated by a gas relieve valve, venting the created argon gas to the outside air. The liquid argon entering the cryostat has been passed through the liquid purification system before entering the cryostat to diminish the oxygen and water content. In the middle

IOP Conf. Series: Materials Science and Engineering 1240 (2022) 012118 doi:10.1088/1757-899X/1240/1/012118



Figure 3: Purity levels in the NP04 cryostat gas volume during the room temperature purification cycle

of the filling cycle the purification system is re-generated to assure that also the second batch of argon enters the cryostat with the lowest possible argon level.

Once the argon level in the cryostat has arrived at the desired cryostat level (argon content of the cryostat is about 840 tons), the cryogenic system as defined in section 3 is switched on, and the argon bath pressure is regulated to 1.05 bar absolute (corresponding saturated temperature 87.6 K), while a continuous flow of about 7 tons/hour of liquid argon is passed through the liquid purification system. The obtained purity level in the NP04 cryostat is given in Fig. 4. The figure shows the results measured by three purity sensors placed at different heights in the cryostat. The red horizontal lines in this graph correspond to a free electron life time in the liquid argon bath of 3 ms, 10 ms and 20 ms. From the curves

in the graph one can draw the conclusion that the free electron lifetime in the liquid argon bath has over this time period been higher than 15 ms, corresponding to a 10's ppt purity level.



Figure 4: Argon purity level in the NP04 cryostat during the operational period between September 2018 and July 2020.

1240 (2022) 012118

doi:10.1088/1757-899X/1240/1/012118



Figure 5: Temperature gradient in the NP04 liquid argon bath, represented as function of height from the bottom of the cryostat.

The negative peaks in Fig. 4 correspond to periods in which a pollution has entered into the cryostat, or to moments at which the purification of the liquid was temporarily stopped. The large negative peak corresponds to the moment the membrane in the warm gas circulation pump in the signal chimney line ruptured. It took some time before the rupture was discovered during which period a small air flow was moment onwards the warm circulation pump has been taken off line.

Fig. 5 shows the temperature gradient over the cryostat volume, measured as a function of the height in the cryostat, as measured from the cryostat bottom. From the figure one can see that the temperature gradient over this volume is smaller than 5 mK, assuring that the reaction of the detector is not influenced by the temperature of its surrounding argon bath. Figure 6 shows the absolute NP04 cryostat argon gas



Figure 6: NP04 cryostat gas pressure during almost two months of continuous operation

pressure as measured over a period of almost two months. During this period this gas pressure is stable within about 1 mbar corresponding to a stability in the bath temperature within about 10 mK.

	NP02	NP04
	(W)	(W)
Cryogenic System	850	1200
Purification System	1950	1900
Cryostat	6500	6100
Miscellaneous	1500	1500
Total heat-load	10800	10700

#### Table 1: estimated heat-loads into NP02 and NP04 systems

Table 1 gives the estimated heat loads into the NP02 and NP04 cryostats, based on measurement of the argon gas flow coming from the cryostat volume being fed into the condenser. After corrections for the heat load brought by the cables and electronics into the cryostat volumes, one can conclude that a heat-load of about  $9.0 \text{ W/m}^2$  is passing through the cryostat wall, slightly higher than the specified 7.5 W/m<sup>2</sup>. The smaller surface of the proto-DUNE cryostats compared to the final DUNE cryostat shall however be taken into account. The figure for the smaller cryostat structure has a higher influence from the cryostat irregularities such as corners and bottom valve connections. Foreseen modifications to the installation of the cryostat's thermal insulation material will also reduce this figure.

After the operational cycle, the cryostats have been emptied using the pump normally used in the liquid purification cycle. This time however the pumps have pushed the liquid to the outside storage tank, from where the liquid argon has been loaded in trucks removing the liquid argon from the CERN site. The cryostats have been warmed up by the natural heat-load into the cryostat volume over a period of several weeks. This time could be taken, since the next cool-down of the cryostat systems was only foreseen in more than a year time, giving sufficient warm up time.

## 5. Conclusion

Two DUNE prototype cryostats and their cryogenic system have been designed, constructed, installed and operated at CERN. During the operational periods of these two cryostats it was proven that the temperature gradients over the cryostat volume and the temperature gradients in time were will within the predefined maximum values, while it also has become clear that the purity level in the cryostats was also well within the acceptable limits. The heat load through the cryostat walls per square meter of cryostat wall was slightly higher than expected. Proposed modifications to the installation of the cryostat's insulation material shall however reduce this number.

Based on the measurement made on the two proto-DUNE cryostats one can conclude that the proposed cryostat system as well as the proposed cryogenic and purification systems are qualified candidates for the future DUNE experiment.

#### 6. References

[1] B. Abi et al. Dune Far Detector Technical Design Report Volume IV. The DUNE far detector single phase technology, 2020 JINST **15** T08010 (<u>https://iopcience.iop.org/article/10.1088/1748-0221/15/08/T08010</u>)

[2] B. Abi et al. DUNE Far Detector Technical ; Design Report Volume I. Introduction to DUNE, 2020 JINST **15** T08010 (<u>https://iopscience.iop.org/article/10.1088/1748-0221/15/08/T08008</u>)

[3] D. Montanari, et al. Development of membrane cryostats for large argon neutrino detectors. 2015 IOP Conf. Ser.: Mater. Sci. Eng. 101:012049

[4] J. Creus et al. Design, construction and commissioning of the proximity cryogenics systems serving two large-scale prototypes of the future DUNE neutrino detector. 2019 IOP Conf. Ser.: Mater. Sci. Eng. 502:01101

1240 (2022) 012118

[5] D. Montanari et al. Status of the LBNF Cryogenic System. 2017 IOP Conf. Ser.: Mater. Sci. Eng. 1757-899X/278/1