NICOLHy - Novel Insulation Concepts For LH2 Storage Tanks

Project deliverable

D3.1 Test setup

Project duration	January 2024 – December 2026
Contractual date of delivery	30/06/2024
Date of delivery	28/06/2024
Reporting class	Public (PU)
Editors:	Finn Reinhard Harwege (BAM) Robert Eberwein (BAM)
Contributors	NA
Deliverable Status	Approved

Co-funded by the European Union

Clean Hydrogen Partnership

NICOLHy project No. 101137629 is funded by the European Union. Views and opinions expressed are however those of the author(s) only and do not necessarily reflect those of the European Union or Clean Hydrogen JU. Neither the European Union nor the granting authority can be held responsible for them.

Document Revisions:

Version	Date	Editor	Overview
0.0	24.05.2024	Finn Reinhard Harwege	First Draft
0.1	26.06.2024	Robert Eberwein	Review
0.2	28.06.2024	Finn Reinhard Harwege	Revision

Approvals:

Version	Date	Name	Organization
1.0	28.06.2024	Robert Eberwein	BAM

The NICOLHy Consortium

Participant No.	Short name	Country
1 (Coordinator)	BAM	Germany
2	UniBo	Italy
3	DLR	Germany
4	NTNU	Norway
5	NTUA	Greece

Abstract

This deliverable presents the current stage of development for a test rig that will be used to conduct thermal conductivity tests of vacuum insulation panel (VIP) systems in cryogenic conditions. An introduction into the measurement of thermal performance in insulation systems is given and standards for the testing of cryogenic insulation and vacuum insulation panels reviewed. The conceptual design of the test rig as a guarded flat plate calorimeter is shown, design considerations are described and an outlook into ongoing and future work is provided.

Content

Abbreviations:

ASTM	**A**merican **S**ociety for **T**esting and **M**aterials
ATEX	**AT**mosphères **EX**plosives
BAM	**B**undes**a**nstalt für **M**aterialforschung und -prüfung
DLR	**D**eutsches **L**uft- und **R**aumfahrt **I**nstitut
DIN	**D**eutsches **I**nstitut für **N**ormung
EN	**E**uropean **N**orm
ISO	**I**nternational **O**rganization for **S**tandardization
LH2	**L**iquefied **H**ydrogen
LN2	**L**iquefied **N**itrogen
NA	**N**ot **A**pplicable
NICOLHy	**N**ovel **I**nsulation **Co**ncepts For **L**iquefied **Hy**drogen Storage Tanks
NTNU	**N**orges **T**eknisk-**N**aturvitenskapelige **U**niversitet
NTUA	**N**ational **T**echnical **U**niversity of **A**thens
PPE	**P**ersonal **P**rotective **E**quipment
PU	**Pu**blic
TTS	**T**echnical **T**est **S**ite
UniBo	Alma Mater Studiorum - **Uni**versita Di **Bo**logna
VIP	**V**acuum **I**nsulation **P**anel
WP	**W**ork **P**ackage

1 Introduction

Within the NICOLHy project novel insulation concepts will be developed and benchmarked. WP3 deals with the tests of these concepts. In this document 'D3.1 Test setup' the requirements and the design for a test rig are described, which allows testing of novel insulation concepts at relevant conditions.

The goal of thermal insulation on a tank for a cryogenic fluid such as liquid hydrogen (LH2) is to reduce the heat flow from the environment into the stored liquid hydrogen. The aims of the test rig are:

- To determine the heat flow generated by prototypes of the novel insulation system and to calculate the thermal conductivities,
- To test installation procedures,
- To test the system behaviour.

The effectiveness of an insulation system can be expressed as the effective thermal conductivity k_e in [W/(mK)]:

$$k_\mathrm{e} = \frac{\dot{Q}x}{A_\mathrm{e}\Delta T} \qquad (1.1)$$

With heat flow $\dot{Q}$ in [W], the temperature difference ΔT in [K] between the warm side and the cold side of the insulation, A_e in [m²] which represents the effective area of heat transfer and x in [m] which is the thickness of the insulation.

In general, the insulation system's effective thermal conductivity is a function of many factors including temperature, conductivity of ambient gases, pressure of ambient gases, mechanical pressure, thickness and more. To obtain values of k_e that are applicable for large LH2 tanks, the test rig should provide an adequately low temperature and a representable size. The results of tests can be used to:

- Compare different designs. During the project, the influence of different parameters (e.g., size of panels, number of layers, stacking patterns, material combination etc.) on the thermal performance must be determined to find optimal solutions.

- Validate numerical models of the insulation system. Numerical simulations of the insulation system will be carried out in WP2, experimental results will be necessary to determine the accuracy of those simulations and to verify assumptions.

- Determine the sensitivity of the thermal performance to changes in conditions. Examples of conditions that should be explored include different atmospheric gases and mechanical load on the panels (gravitational or external, for example created by a mounting system)

- Simulate the change in system thermal performance due to a component failure (e.g., loss of vacuum in a vacuum insulation panel).

- Determine the viability of the novel insulation design by comparing the thermal conductivity as measured on the test rig to the thermal conductivity of conventional cryogenic insulation systems.

In addition to testing the thermal performance, the test rig will help to gain insights into the mechanical performance of the insulation system. Deformation or damage to the vacuum insulation panels due to thermo-mechanical and mechanical loads can be observed or measured. As a by-product, practical challenges of assembling the system and general handling of the vacuum insulation panel (VIP) will also be observed while carrying out the experiments.

2 Heat flow determination

Equation 1.1, shows that to determine the thermal conductivity, in addition to measuring the dimensions of the tested insulation, a known temperature difference between the cold and hot sides of the insulation must be generated and the heat flow measured. This section presents existing standards and methods for determining the performance of insulations applicable to cryogenic storage tanks and for VIPs.

2.1 ASTM C1774: Standard Guide for Thermal Performance Testing of Cryogenic Insulation Systems

This standard gives information on the laboratory testing of thermal insulation systems at cryogenic temperatures. It presents guidelines on the measurements to be carried out, calculations, and reporting of thermal conductivity, as well as presenting multiple examples of test apparatus designs.

Two kinds of apparatuses are introduced. In absolute apparatuses, the design is such that the heat flow measured is exclusively through the insulation specimen that is tested, while in comparative apparatuses, there may be an additional (but constant) heat leak from the environment that is also measured. As the name implies, the thermal conductivity values from an absolute apparatus can directly be used, while a relative apparatus by itself can only provide comparative testing. By testing an insulation system first in an absolute apparatus, and afterwards in a relative apparatus, the relative apparatus can be calibrated to provide thermal conductivity values that are usable as absolute values.

Additionally, two methodologies for measuring the heat flow are introduced: Electrical power and boil-off calorimetry. With electrical power testing, the heat flow is determined by measuring the power consumed by a cryocooler to keep a steady temperature difference between the cold side and hot side. With boil-off calorimetry, the cold side boundary is set with a cryogenic fluid in a test tank. The mass flow of fluid that evaporates (boil-off rate) is measured and with the knowledge of the latent heat of evaporation of the fluid at the pressure inside the test tank, the heat flow can be calculated.

2.2 ISO 21014:2019: Cryogenic vessels - Cryogenic insulation performance

ISO 21014:2019 focusses on the practical determination of the heat leak into cryogenic vessels to determine the insulation performance, by determining the loss of mass by boil-off at standard conditions. From these measurements, the percentage loss of product and the holding time can be determined.

2.3 DIN EN 17140: Thermal insulation products for buildings – Factory-made vacuum insulation panels (VIP)

DIN EN 17140 specifies characteristics and testing of VIPs for the thermal insulation of buildings. Thermal performance is measured in guarded hot plate or heat flow meter apparatuses at conditions close to standard conditions. Measurements at the center of panels and measurements including a joint between two panels are then combined to determine rated effective system thermal conductivity, where a safety factor is also included. Methodologies of artificially aging the panels and determining the deterioration of their thermal performance are also specified.

Additionally, the testing of mechanical properties and fire resistance for VIPs is included in the standard.

2.4 Evaluation of the applicability of standards

Since the insulation system proposed in NICOLHy is novel, the applicability of the existing standards requires critical evaluation. Since during the project duration of NICOLHy, no complete tank with the novel insulation concept will be built, ISO 21014:2019 is not applicable and was only included in the previous section for completeness.

ASTM C1774 is generally applicable with some adaptations. The standard is only applicable to double-walled systems with insulation placed in between and with a primarily homogeneous structure of the insulation material. The standard is therefore suitable for determining the thermal conductivity of materials that are combined with a vacuum or a gas. NICOLHy is aimed for single-walled systems with insulation installed on its outer side, which makes the direct application of the standard unsuitable. However, the measurement systems and design principles of ASTM C1774 are applicable. When defining the tests, some additional considerations must be made to adequately take into account the effects of the joints between the panels, i.e. to arrange them in such a way that the tested section is representative of the whole system.

The thermal testing procedures specified in DIN EN 17140 do not include the relevant temperature range for a cryogenic tank and it only considers a surface covered with a single layer of panels, not stacked layers as proposed in NICOLHy. Nevertheless, the calculation of system thermal conductivity based on thermal performance at the center of the panel and the effect of joints could be a potential base for doing similar extrapolations. Additionally, some of the mechanical and fire resistance tests specified may be applicable for the NICOLHy project.

3 Conceptual Design

Based on the goals of testing outlined in section 1, the general concept for the test rig was chosen to be a guarded flat plate boil-off calorimeter that operates with liquefied nitrogen (LN2). LN2 is used to keep the flat plate at a low temperature for a long time. LN2 boiling point is 77 K which is higher than LH2 with 20 K. This change of test fluid brings several advantages for testing. First, LN2 has a higher density and a similar evaporation enthalpy compared to LH2, from this perspective longer tests with a much smaller test rig can be arranged. Second, LN2 is less dangerous compared to LH2, because it is less cold and not burnable. This reduces costs for ATEX, costs for the equipment to handle LH2, and costs for the test preparation and execution. Third tests with LH2 requires a higher safety level for planning the test rig and for performing the tests which hinders to investigate some aims of the NICOLHy project. For these reasons, it is useful to do the tests with LN2 instead of LH2.

The proposed scale of the test rig includes a square test surface with sides of 2.5 m, on a test-tank, which is embedded in a guard-tank. The guard tank extends the surface by 0.25 m per side to the test surface which creates in total a cold plate of 3 m x 3 m. The primary restrictions for the maximum feasible size are the manufacturability and transportability of the test apparatus.

The test-tank is surrounded from 5 sides by the guard-tank. Both tanks contains LN2 at its boiling temperature, so they stay at an equal temperature and there is no heat flow through these 5 sides. The sixth side is the test surface for insulations. Therefore, any heat flow into the test-tank must pass through the insulation applied to the test surface.

The guard-tank is embedded in a shell that contains its insulation. This insulation based on a combination of a powder and a vacuum as it is typically applied for LNG tanks. This insulation is necessary to prevent excessive boil-off, which reduces costs and the overall size of the test-rig. The sixth side of the guard-tank, which is not in contact with the test-tank or the insulation, is the guard surface, which is also covered with the test insulation during a test.

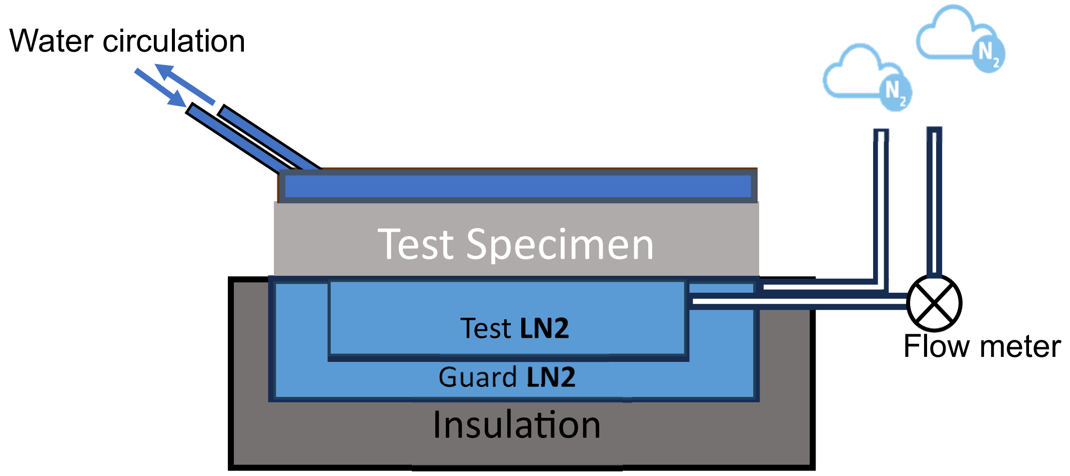


Figure 1: Schematic of the test rig (cut view)

To ensure steady boundary conditions for the test insulation, the warm side of the insulation, is covered by a water-supported heat exchanger. The water circulates between the test rig and a large water underground reservoir on site, which ensures a constant temperature over the entire test duration.

The boil-off line from the guard-tank is left open to the atmosphere, while during the test and measurement of thermal conductivity. The boil-off from the test tank is routed through a flow meter to be measured. The boil-off lines from both tanks will be vented to the atmosphere at an appropriate height to the environment to not pose any asphyxiation risk.

Using the mass flow rate $\dot{m}$ in [kg/s] measured by the mass flow meter, the heat flow $\dot{Q}$ into the test-tank in [W/s] can be calculated:

$$\dot{Q} = \dot{m} h_{\mathrm{fg}} \qquad (3.1)$$

with the latent heat of evaporation h_{fg} of liquid nitrogen in [J/kg] at the pressure inside the test-tank. It is assumed that no heat is transferred from the tank to the gaseous nitrogen, which according to ASTM C1774 only results in a negligeable error if the ullage space is small.

3.1 Sensors and Instrumentation

To measure the thermal conductivity of the tested insulation, the temperatures at both sides of the test specimen and the mass flow rate of nitrogen from the test-tank need to be known. This can be achieved through simple temperature measurements, for which thermocouples will be used and the flow meter on the test-tank boil-off line

Additionally, sensors will be added to monitor the condition and operation of the test rig:

- A pressure sensor on the bottom of each tank will be used to determine fill level through the hydrostatic pressure. Knowledge of the fill level will be of obvious importance during the filling of the tank, but also to ensure that the conditions are as similar as possible from test to test.

- A pressure sensor in the outer insulation will allow the monitoring of the condition of the vacuum. This will support maintenance decisions such as indicating the need for re-pumping vacuum due to leaks.

- Temperature and pressure sensors may be installed in the vent tubes to obtain data for a deeper understanding on the operation of the test rig.

Aside from the sensors installed on the test rig, further sensors may be installed on the tested insulation, depending on the purpose of the test. Examples include strain gages and temperature sensors, but a careful investigation will be needed to determine the influence that these sensors may have on the insulation systems behaviour.

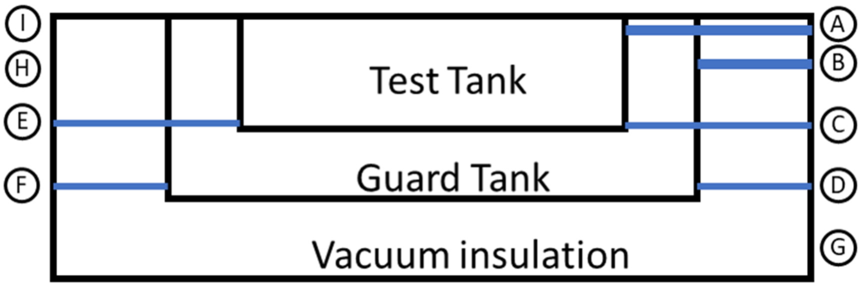
The target for the project is an insulation system with a minimum heat leak of $q_{min} = 1\,\mathrm{W/m^2}$, which on the 6.25 m² test area would result in a heat flow of $Q_{min} = 6.27\,W$. Inserting this into equation 3.1 and multiplying with the density of gaseous nitrogen at standard conditions yields $\dot{V}_{min} = 1{,}51\,l/min$. For the measurement of the boil off mass flow rate, a Driesen-kern TSI5300 flow meter will be used. It was chosen for its high accuracy and its range of 0.05 – 300 l/min for nitrogen at standard conditions, which is adequate to measure the range of thermal conductivity that is expected to be tested on the test rig. Should the sensor range unexpectedly not suffice, it is possible to add a second sensor in parallel to extend the measurable range of heat flows. The accuracy of the sensor is 2% of reading or 0,05 l/min (higher value counts), which results in a 3.3% error at $\dot{V}_{min}$.

3.2 Connection lines

Additional to the vent tubes and lines for sensors mentioned in the previous sections, the test and guard-tank need a tube for filling the tanks with LN2. All lines that are connected to a cold side (the tanks) and a warm side (the outside of the insulation), must deal with thermal deformations.

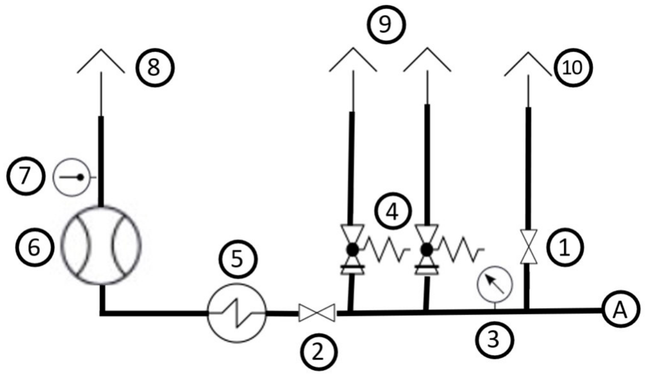
Careful consideration must be taken to minimize the thermal bridges that connection lines create. Running lines on the test-tank through the guard-tank prevents heat leaks into the test-tank, but heat leaks into the guard tanks should be minimized. For this, gas traps and keeping cross sections of tubes as small as possible represent proven methods.

Figure 2 depicts all lines and interfaces on the test rig. The attached connection line on the test-tank vent (A) is the most complex and therefore depicted in this report. A bypass valve (1) enables to open the test-tank to the atmosphere, this is intended to be used during filling and initial cooldown. When this phase is completed and the measuring is started, the bypass valve is closed and the valve to the flow meter is opened. A heat exchanger ensures that no LN2 or cold vapour comes into contact with the sensor and the flow through the sensor is within the required operating temperature range. The redundant safety valves (4) ensure that the pressure inside the tank cannot increase above the specified operating pressure, even if the bypass valve and/or the valve to the mass flow meter is closed.



A	Test tank vent
B	Guard tank vent
C	Test tank fill
D	Guard tank fill
E	Test tank pressure sensor
F	Guard tank pressure sensor
G	Vacuum port
H	Vacuum Gauge port
I	Burst disc

Figure 2: Schematic of the connection lines at the test rig



1	Bypass valve
2	Sensor valve
3	Temperature sensor
4	Safety valves
5	Heat exchanger
6	Flow meter
7	Temperature sensor
8	Sensor vent
9	Safety valves vent
10	Bypass vent

Figure 3: Schematic of the test tank vent line

3.3 Thermal design

It is important to provide the test surface with a uniform temperature, as this is one of the inherent assumptions for equation 1.1. The test surface will be equipped with stainless steel elements that stay in contact with LN2 at all fill levels in the test-tank. This brings the test-surface into contact with the LN2 on several locations and ensures a uniform temperature boundary condition.

The other objective of the thermal design is to decrease the total heat capacity of the test rig. First estimations on preliminary designs of the test rig show, that during the cooldown from ambient temperature to test conditions of the test rig and test insulation, needs an evaporation of approximately 0.9 m³ of LN2. This is not only a significant driver of the cost per test, but a higher thermal capacity of the test rig also increases the necessary time for it to cool down which increases the time per test.

Since the two objectives of minimizing heat capacity and providing a uniform temperature on the test surface are competing, the design of the stainless-steel elements will be determined through numerical studies to find an optimal solution.

3.4 Mechanical design

The test rig will be a welded construction, with most parts built from flat sheet metal. The material that the test rig is constructed from needs to remain ductile in cryogenic conditions. Austenitic steels are commonly used in cryogenic service and will be used for the test rig. 304L stainless steel is chosen due to its availability, weldability, and cost.

The difference in temperature between the tanks (at approximately 77 K) and the outer surface of the insulations (at approximately ambient temperature, around 295 K) causes thermal stresses in the material connecting those sections. Stainless steel contracts about 3 mm/m when cooled from standard temperature to 77 K. With the target dimensions of a 3x3 m² cold plate, the connecting section between the insulation walls and the guard tank must allow a movement of 4.5mm in each direction. To enable this movement, some compliance must be designed into these sections, while keeping the vacuum insulation layer gas tight. To enable this, corrugated panels will be used, that are more flexible than flat sheets when tensioned perpendicular to the folds. Some plastic deformation is likely to occur, especially in the corners, but this can be accepted.

Aside from maintaining the mechanical integrity to prevent leakage, another design requirement is maintaining the flatness of the test surface. This can be ensured by reinforcing the test tanks with baffles. These baffles will also be designed such that they help to create a uniform temperature on the test surface. Figure 4 shows a preliminary drawing of the test rig design.

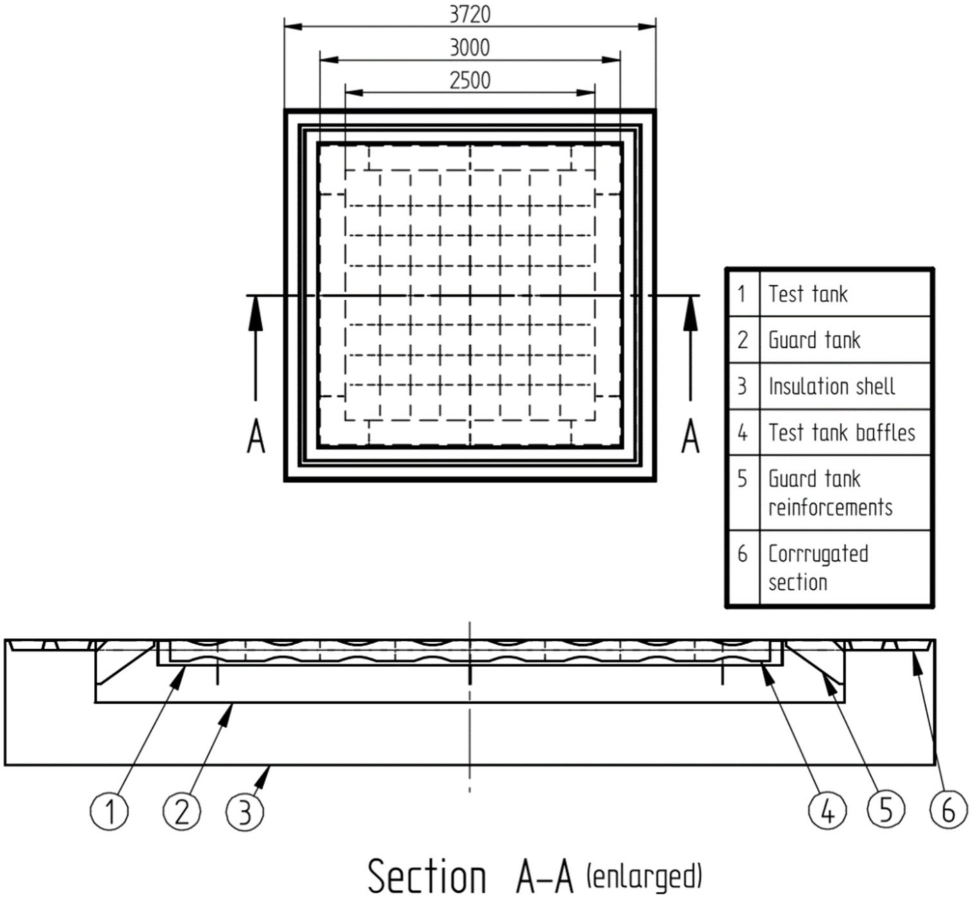


Figure 4: Sketch of the test rig

Figure 5 shows a screenshot from the ongoing structural FEM-analysis on the corrugated sections. The boundary conditions simulate the thermo-mechanical strain created from the cooldown of the tanks, with the goal to choose a profile for the sheet metal that minimizes plastic deformations. In this model, the influence of the vacuum pressure acting on these sections will also be investigated. Based on those results, promising designs will be tested with a coupled thermal and structural analysis.

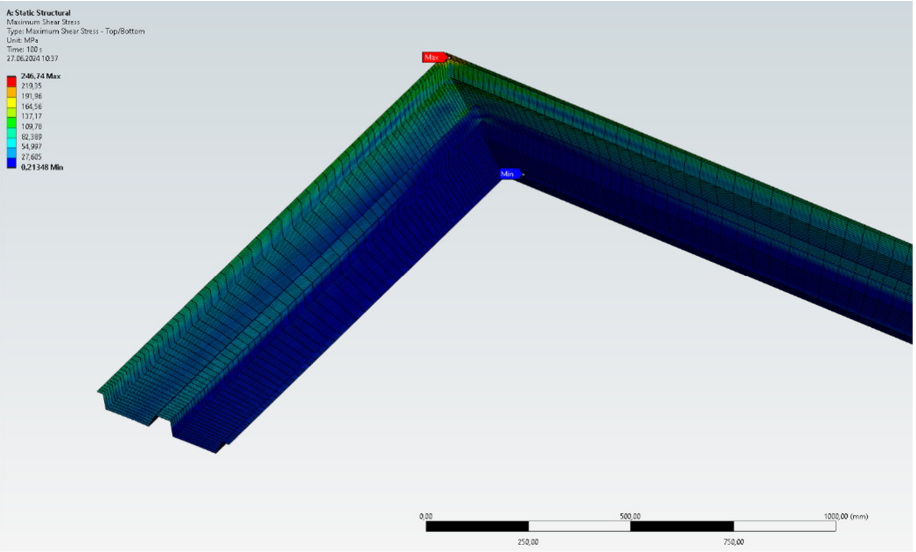


Figure 5: FEM analysis of corrugated section

3.5 Safety considerations

The test rig must be safe to use under all operating conditions. Aside from the safety risks from the handling of cryogenic fluids and nitrogen as an asphyxiant. This will be minimized with Personal Protection Equipment (PPE) and appropriate protocols.

An important concern is preventing over-pressurization of the tanks. The tanks will be designed to operate by overpressures less than 0.5 bar, so that they are not considered pressure vessels according to the Pressure Equipment Directive 2014/68/EU. This requires that vent lines and safety valves provide sufficient mass flows. Since the mass flow from the guard-tank does not need to be measured, as long as the boil-off lines are adequately sized, pressure buildup can be prevented without the use of safety valves.

The implementation of a mass flow meter with a small cross section on the test-tank vent dictates the necessity to use safety valves to ensure that in the case of pressurization, the valves open and the boil off gases can vent at a sufficient flow rate. Figure 3 shows the position of the safety valves in the system. ISO 4126 and BS EN ISO 21013-3 are used for the sizing of the safety valves and the identification of critical cases.

The first situation that requires investigation is the filling procedure. During filling, the test or guard-tank is connected to another storage tank with higher pressure or a pump that generates a pressure in between. During the initial stage of filling a warm tank, the injected LN2 will almost instantly evaporate, expand by a factor of approximately 700 to its gaseous form, and cool the walls. Due to this large expansion ratio, even a small volumetric flow of LN2 can produce a large volumetric flow of nitrogen vapour that needs to be vented from the tanks, until the tank walls are cooled closer to 77K. Therefore, the mass flow into the tank needs to be strongly restricted in the initial cool down period, either by means of a pressure reducer set to the maximum operating pressure of the tank, or by careful modulation of the fill valve while monitoring the pressure in the tank. In addition, this method is useful to maintain structural integrity which is affected by thermal loads and stresses, and which can be achieved by slow cooling of the entire test rig.

The scenario that has been determined to be critical for the design of the safety valves and vent lines is the loss of insulation. An increase in heat flow into the tank results in an increase in boil off. The maximum mass flow will be reached when the insulation is performing worst. For the test tank, the critical case that produces the highest heat flow is an empty guard tank with no insulation installed on the test surface, for the guard tank the worst case, is an empty test tank with no insulation installed on the test surface and a loss of vacuum on the insulation layer.

Another scenario that needs to be considered is a leak of LN2 from the guard tank into the insulation. The nitrogen will not only decrease the insulation performance, it will also evaporate, and expand, which creates an overpressure in the previous vacuum insulation. Therefore, a burst disk will be integrated that ventilates the insulation before it reaches an unacceptable overpressure.

3.6 Location

The test rig will be installed and used at the BAM TTS test site for technical safety in Horstwalde. A suitable place has been chosen that provides access to the necessary electrical power, network connection, and the water that will be used to ensure the steady state warm side boundary condition. There is also access for trucks delivering the LN2 required for the tests.

To further limit the influence of environmental conditions, a tent will be constructed covering the test rig. The tent can also be removed, to realize a more “realistic” boundary condition with changing solar radiation and temperature.

Figure 6: Test location at BAM TTS

4 Conclusions

In this first phase of the project, the general concept of the test bench was finalized after a review of the standards and the state of the art. ASTM C1774 was found to be the standard applicable for the large-scale tests within the NICOLHy project. The chosen operating principle of a flat plate boil-off calorimeter is well established, but the necessary scale for testing during this project is new.

Critical design challenges for the test rig were identified and principles for solving them were developed. Ongoing work is the exact design of the tank structure and specification of components based on the requirements established in section 3.

Future work will involve the testing of single components (such as core materials that can be used in the vacuum panels), which will aid design decisions in other work packages. Additionally, the manufacturing of prototype vacuum insulation panels and development of techniques for this is necessary. Once those prototypes are created, testing of their mechanical and thermo-mechanical behaviour can be carried out at BAM.

5 Literature

ASTM (2013). ASTM C1774 Guide for Thermal Performance Testing of Cryogenic Insulation Systems. ASTM International. https://doi.org/10.1520/c1774-13r19

ISO (2019). ISO 21014:2019-10 Cryogenic vessels - Cryogenic insulation performance. International Organization for Standardization

DIN (2021). DIN EN 17140:2021-08, Wärmedämmstoffe für Gebäude_- Werksmäßig hergestellte Vakuumisolationspaneele (VIP) - Spezifikation; Deutsche Fassung EN_17140:2020. Beuth Verlag GmbH. https://doi.org/10.31030/3269172