

The feasibility and application of multi-layer vacuum insulation for cryogenic hydrogen storage

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Dissertation submitted in partial fulfilment of the requirements for the degree of
Masters in Engineering
of the North-West University at the Potchefstroom Campus

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Potchefstroom

2011

ABSTRACT

A need was identified to test multi-layer vacuum super insulation (MLVSI) used in cryogenic applications for hydrogen storage. The study focuses on the application of commercially available MLVSI to a locally patented liquid hydrogen cryogenic storage system. This led to an investigation of different types of multi-layer vacuum insulation configurations, as well as further research on tank inlet coupling configurations. It includes the manufacturing of a liquid nitrogen testing cryostat to be able to test and evaluate the system performance.

The first set of tests was based on the development of an inlet coupling configuration to limit heat transfer through the inner tank inlet, of a double cryogenic tank system in order to reduce gas boil-off. The couplings were manufactured in the form of a bellow to handle cryogenic vacuum levels, while ensuring low heat transfer rates between inner and outer tanks. It was found that various coupling designs can be considered to limit gas boil-off.

The second set of tests was conducted on a specific MLVSI configuration to determine its effectiveness to insulate the spherical header surface of a typical hydrogen storage vessel. The installation procedure, to limit heat transfer and boil-off due to edge effects in this configuration was investigated. It was found that insulation-overlap-edge effects will always have an impact on insulation performance when a spherical header of a storage vessel is insulated, due to its specific geometry. A time efficient way to install MLVSI on such a spherical header is presented and evaluated.

Further investigations were carried out by combining findings into one single system to determine the performance of an optimised insulated cryogenic system. It was found that copper plate discs installed between the vanes of a bellowed inlet/outlet nozzle is the most promising to limit heat transfer to the cryogenic fluid.

Keywords: multi-layer, vacuum insulation, super insulation, cryogenic, hydrogen storage.

DECLARATION

I, Jacobus Henry Hodgman (Identity Number: 8612225059082) hereby declares that the work contained in this dissertation is my own work. Some of the information contained in this dissertation has been gained from various journal articles; text books etc, and has been referenced accordingly.

Initial & Name

Witness

ACKNOWLEDGEMENTS

Special thanks to all the people and companies involved making this project possible. To Prof. Johan Markgraaff who stood by me during the fulfilment of this project as my supervisor, my special thanks. I would also like to thank the North West University for their facilities, whose assistance ensured that this project is successful, and lastly, a special thanks to HySA (DST Hydrogen S.A.) for the financial support.

CONTENTS

List of Figures	vi
List of Tables	ix
Abbreviations	x
Chapter 1: Introduction.....	1
1 General.....	1
1.1 Problem Statement	4
1.1.1 Aim	4
Chapter 2: Literature Survey	5
2 MLVSI Components.....	5
2.1.1 Shield Material	5
2.1.2 Spacer Material.....	7
2.1.3 Film Material	9
2.2 Common Behavior of MLVSI.....	9
2.3 Testing Methods for MLVSI.....	12
2.4 MLVSI Installation	14
2.5 Summary	15
Chapter 3: Experimental Design	16
3 Introduction.....	16
3.1 Experimental Cryogenic System	16
3.2 Operational Requirements	18
Chapter 4: Detail Design	20
4 Background	20
4.1 Cryostat Externals.....	21
4.1.1 Cryostat Outer Tank.....	21
4.1.2 Vacuum Pipeline	23
4.2 Cryostat Internals.....	24
4.2.1 Cryostat Inner Tank	24
4.2.2 Inlet Coupling/Nozzle	25
Chapter 5: Manufacturing and Assembly.....	28
5 Introduction.....	28
5.1 Inner Tank and Coupling Manufacturing	28
5.2 Application of MLVSI.....	33
Chapter 6: Experimental Setup and Procedure	35

6	Experimental Setup	35
6.1	Configuration Testing Procedure.....	39
6.2	Assembly Testing.....	41
6.2.1	Inner Tank Coupling Testing	43
Chapter 7: Assembly Modifications		47
7	Background	47
7.1	Coupling Modifications.....	47
7.1.1	Coupling Modification A	47
7.1.2	Coupling Modification B	51
7.1.3	Coupling Modification C	52
7.1.4	FEM Coupling Steady State Simulation	54
7.2	MLVSI Application Modifications	58
7.2.1	Elimination of Edge Effects by Changing Tank Geometry	58
7.3	Discussion	60
Chapter 8: Optimum System		62
8	Introduction.....	62
8.1	Optimum System Performance	62
Chapter 9: Conclusions.....		66
9	Bibliography.....	68
Appendix A: Theoretical Model		72
Appendix B: Safety		74
Appendix C: Calculations		76
Appendix D: Experimental Data		79
Appendix E: Part List and Suppliers		83
Appendix F: Design Drawings		84

List of Figures

Figure 1: Hydrogen density vs. temperature at certain pressures, modified after [6].....	2
Figure 2: Illustration of MLVSI layers	5
Figure 3: Absorption and reflection processes associated with a non-transparent medium, modified after [9]	6
Figure 4: Spacer Matrix Configurations: a) randomly orientated, b) parallel structure c) staggered beam structure [10]	8
Figure 5: Illustration of liquid nitrogen calorimeter for testing the effectiveness of MLVSI, modified after [17]	13
Figure 6: Illustration of a boil-off calorimeter for testing MLVSI, modified after [17]	14
Figure 7: Schematic illustration of the required experimental cryogenic system	17
Figure 8: Transparent CAD model of the experimental system	20
Figure 9: Schematic of the cross section view of the O-ring gland flanges	22
Figure 10: CAD model exploded view of the cryogenic system outer tank	22
Figure 11: Exploded view of the CAD model of the vacuum pipeline	23
Figure 12: Exploded view of the CAD model of the experimental system internal components	24
Figure 13: CAD model illustrating the heat path through a thin walled bellow	26
Figure 14: Inner tank dome male and female die manufacturing	28
Figure 15: Inner Tank Male and Female Dies.....	29
Figure 16: Inner tank dome pressing process.....	29
Figure 18: Cross sectional schematic view of the redesigned female die	30
Figure 17: Manufactured dome indicating buckling	30
Figure 20: Illustration of the cryogenic inner tank mounted to a stand to facilitate MLVSI installation.	31
Figure 19: Manufactured 150mm Ø domes from various billet sizes.....	31
Figure 21: Photo of the assembled and weld-joined inner coupling provided with flanges	32
Figure 22: Illustration of MLVSI cutting using a jig over-lay on the insulation with a newspaper page as intermediate layer	33
Figure 23: A single layer of MLVSI ready to be installed to the inner tank	34
Figure 24: Illustration of the folding process in order to limit edge effects.....	34
Figure 25: Photo of the experimental cryogenic system for measuring the performance of MLVSI around a vessel with spherical shaped end caps.....	35
Figure 26: E2M18 and E1M18 vacuum pumps performance chart	36
Figure 28: Thyracont high vacuum sensor and control unit.....	37
Figure 27: E2M18 vacuum pump experimental performance chart.....	37
Figure 29: Schematic of temperature measurement positions	38
Figure 30: Photo of the temperature analogue interface	38

Figure 31: Schematic of the PWR high precision bench scale used to measure system weight loss (boil-off)	39
Figure 32: System weight vs. time indicating an increased boil-off weight for old insulation	41
Figure 33: System weight vs. time illustrating deviations between alternative system setup tests	42
Figure 34: System weight vs. time for a conventional inlet pipe.	43
Figure 35: Photo of the coupling installed with top and bottom thermocouples shown	44
Figure 36: System weight vs. time measurement for the bellowed coupling	44
Figure 37: Liquid nitrogen boil-off rates for bellowed coupling and inlet pipe	45
Figure 38: Graph of the temperature differences between the “hot” and “cold” sides of the inlet pipe and the bellowed coupling configuration	45
Figure 39: MLVSI surface temperature	46
Figure 40: A CAD model of the assembled Coupling A	47
Figure 41: Exploded view of the CAD model of Coupling A	48
Figure 42: Heat flow path through modified column support	49
Figure 43: Photo of the manufactured and assembled coupling A	50
Figure 44: Photo of the manufactured and assembled coupling B	51
Figure 45: A schematic illustration of a ZAL 45 part for coupling C to limit radiation	52
Figure 46: Photo of the manufactured and assembled Coupling C with wrapped MLSVI to limit radiation to the bellow.	52
Figure 47: FEA result of the heat flux through Coupling B	55
Figure 48: FEA of the steady state temperature distribution (°C) through modified couplings: a) stainless steel columns and b) glass fibre columns	56
Figure 49: Temperature distribution through Coupling B for various copper heat sinks positions; a) Evenly spaced, b) evenly spaced on the high temperature side, c) evenly spaced on the low	57
Figure 50: Exploded view of a CAD model of the inner tank and the ZAL45 insulation end caps	58
Figure 51: CAD model illustration of end cap and MLSVI installation to a pressure vessel	59
Figure 52: System weight vs. time for folding and end cap configurations respectively.....	59
Figure 53: System weight vs. time for different coupling configurations tested	60
Figure 54: Model of optimum coupling	62
Figure 55: Photo of the manufactured and assembled optimised coupling (Coupling D)	63
Figure 56: Temperature difference over the optimised coupling (Coupling D)	63
Figure 57: Optimised System Weight vs. Time Measurement.....	64
Figure 58: Evaporated mass (boil-off) for ten minute increments vs. time showing how steady state conditions is reached	65
Figure 59: Thin-walled insulated nitrogen tank	72
Figure 60: Multi-Layer Insulation Wrapping Machine (NASA) [25]	73
Figure 61: Fabricating Apparatus for Multilayer Insulation Blankets [29]	73

Figure 62: Gas Boil-off vs. Insulation Thickness.....	77
Figure 63: Gas Boil-off vs. Tank Radius	77
Figure 64: Top and bottom coupling temperatures vs. time for inlet pipe.....	79
Figure 65: Top and bottom coupling temperatures vs. time for coupling.....	79
Figure 66: Top and bottom coupling temperatures vs. time for improved Coupling A.....	80
Figure 67: System weight vs. time for the coupling compared to the improved Coupling A	80
Figure 68: System weight vs. time for coupling A compared to Coupling B	81
Figure 69: System weight vs. time for coupling B compared to Coupling C	81
Figure 71: Top and bottom coupling temperatures vs. time for ZAL 45 insulation	82
Figure 72: Top and bottom coupling temperatures vs. time for optimum system	82

List of Tables

Table 1: Emissivity of various shield materials [[9]]	7
Table 2: Thermal conductivities of typical MLI sample configurations of materials tested at 13mbar, modified after [12]	11
Table 3: Considered materials to manufacture cryogenic inner vessels	25
Table 4: Thermal properties for components used in the FEM model.....	54
Table 5: ANSYS probe temperatures for heat sink configurations of Coupling B	57
Table 6: Boil-off improvements for modified couplings	60
Table 7: Boil-off rate for various Insulation materials used to insulate a thin walled nitrogen container	72

Abbreviations

CAD	–	Computer Aided Design
CBT	–	Cold Boundary Temperature
CNC	–	Computer Numerically Controlled
CVP	–	Cold Vacuum Pressure
EES	–	Engineering Equation Solver
FEM	–	Finite Element Modelling
MLI	–	Multi-layer Insulation
MLIB	–	Multi-layer Insulation Blankets
MLVSI	–	Multi-layer Vacuum Super Insulation
PMLIB	–	Perforated Multi-layer Insulation Blankets
PPE	–	Personal Protection Equipment
WBT	–	Warm Boundary Temperature

Chapter 1: Introduction

1 General

Fossil fuels are the modern world's primary and most important energy source. The main type of which is oil, used to generate heat for cooking and lighting applications during the beginning of the nineteenth century. Today, it is mostly used as a fuel for internal combustion engines and provides mobility for planes, cars, trains, trucks and boats.

Fossil fuels consist of carbon and hydrogen atoms and by burning these fuels, carbon dioxide and other products are being produced that are harmful to the environment. Scientists believe the amount of fossil fuels that has already been burnt has produced enough carbon dioxide to develop a rapid climate change, and that in the year 2060, the world will be consuming three times the energy of today [1]. Consequently alternative energy sources have to be developed that are cost effective and need to fill the modern world's energy requirements without further increasing carbon dioxide levels. It is believed that hydrogen can be used as an alternative energy carrier for the use in internal combustion engines in order to alleviate the problem [2].

Hydrogen is the lightest gas in the universe with no smell, colour or taste and burns cleanly producing very little or no harmful emissions [3] [4]. Another advantage of hydrogen is that it can be produced by a number of feedstocks like fossil fuels, water and organic matter. Hydrogen has some disadvantages such as high production cost, storage problems and the high explosiveness of the gas which makes it dangerous to work with. Some of the most common processes to produce hydrogen are electrolysis of water, coal gasification and steam reformation.

It is claimed that hydrogen can be stored in four different ways namely: compressed, by liquefaction, physisorption and as an element in compound, metallic hydrides. According to Zhou (2004), compressing hydrogen as a gas is the simplest way to store it up to 20 MPa, however, it is claimed that the energy density is too low for transport

applications. For transport applications the pressure must be at least 70 MPa, which is four times higher than where use is made of internal combustion engines.

Hydrogen is also stored as a liquid at very low temperatures (-253°C) but at high pressures (70 MPa) in cryogenic systems. Hydrogen in liquid phase has a more promising density to be used in transport applications than in gas phase, but faces certain challenges like cost and efficiency of the liquefaction process [5]. The hydrogen density versus temperature at certain pressures is shown in Figure 1.

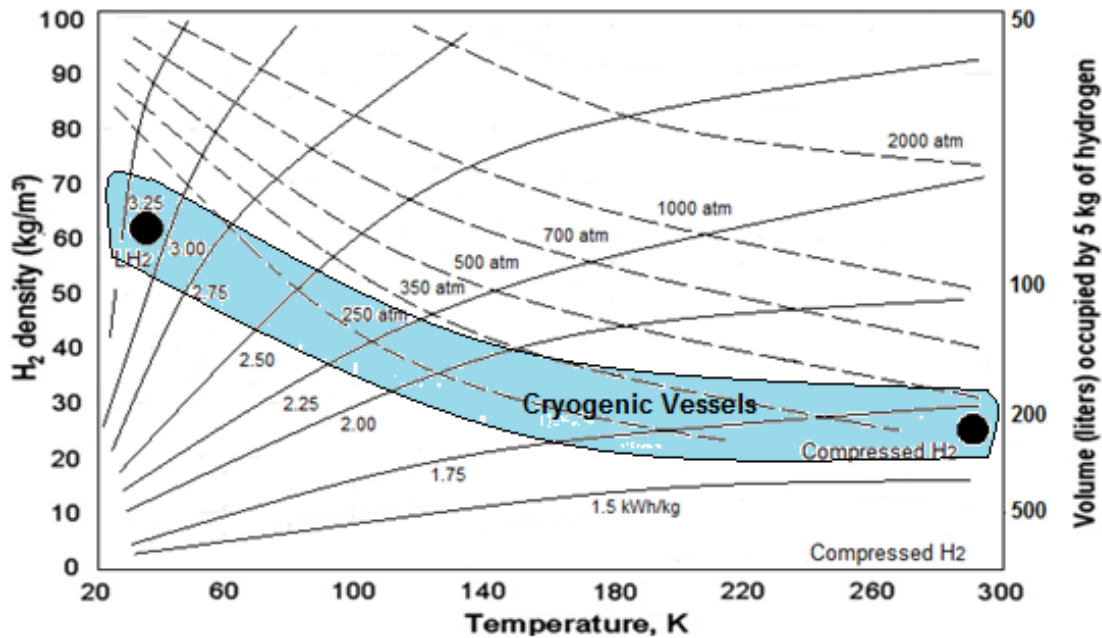


Figure 1: Hydrogen density vs. temperature at certain pressures, modified after [6]

Figure 1 shows that 1.75 kWh/kg of energy is required to compress hydrogen to 350 atm. at ambient temperature. The density is between 20 kg/m^3 and 30 kg/m^3 and it would take a volume of 200 litres to store 5 kg of this compressed hydrogen, whereas a hydrogen density of between 65 kg/m^3 and 70 kg/m^3 can be reached when it is liquefied at a temperature of 20K. However, to liquefy hydrogen cryogenically, 3.25 kWh/kg of energy is required with the advantage that 5kg can be stored in a volume of only 80 litres.

To store liquid hydrogen cryogenically, two times more energy is required than what is required to store compressed hydrogen gas at ambient temperatures but with the added advantage that a density of more than three times that of compressed hydrogen is obtained [6]. Apart from the cost implications to liquefy hydrogen and store it cryogenically this method of storage makes it possible to utilize hydrogen for vehicle applications.

In order to store liquid hydrogen cryogenically, very sophisticated insulation materials and configurations are required to mitigate hydrogen boil-off. This means cryogenic systems must be well-insulated with a highly effective insulation material to prevent the liquid hydrogen loss through boil-off to the atmosphere. Bulk fill insulation is the most common type of insulation and is used in a vacuum space to limit heat transfer due to conduction. Examples of these materials are inorganic compounds in their fibrous forms such as fibreglass and aerogel, perlite powder or silica powder [7].

The above mentioned insulation materials were used in a spherical, thin walled container to demonstrate the insulation effect on nitrogen boil-off theoretically. This thin walled nitrogen container with theoretically calculated boil-off rates for different insulation materials is presented in Appendix A. It is calculated that with a relatively effective insulation material (silica powder in a vacuum) with a thermal conductivity of 0.0017 W/mK the nitrogen boil-off rate is 7 litres per day. Although this insulation has a relatively low thermal conductivity, gas boil-off is still high.

It is believed that the most effective heat barrier for cryogenic applications is layered composite insulation material operating in high vacuum. It is claimed that for cryogenic applications MLVSI is the industry standard for insulating cryogenic containers. This composite insulation material shows much better boil-off results than conventional insulation.

1.1 Problem Statement

Conventional high temperature insulation does not limit and reduce boil-off or the loss of hydrogen to acceptable limits when used for cryogenic storage systems. Use is made of multi-layer vacuum super insulation to mitigate boil-off of hydrogen. Although multi-layer insulation seems to deliver outstanding results the application method and installation is not generally known nor is the feasibility in combination with vacuum, well documented.

1.1.1 Aim

The aim of this study is to review the components of MLVSI and their function and to determine the feasibility of application of MLVSI to a spherical pressure vessel for cryogenic hydrogen storage. It is also the aim to optimize inlet/outlet nozzle configurations to reduce heat transfer to the cryogenic fluid in order to reduce unnecessary loss of hydrogen through boil-off.

Chapter 2: Literature Survey

2 MLVSI Components

Typical multi-layer vacuum super insulation is composed of shield material, film material and spacer material (Fig. 2). This assembly of the combination of materials is employed in vacuum space to limit heat transfer to a cryogenic fluid. Heat can be transmitted in three ways namely conduction, convection and radiation. The shields minimise heat flow due to radiation and the spacer material minimises solid conduction between these alternative shields. Generally, the shield material is plated on a film material to add strength to the insulation for installation purposes. By installing this combination of materials within vacuum space it is possible to further limit heat transfer due to convection. In some applications only shield and spacer materials, without a film material are used in a vacuum space.

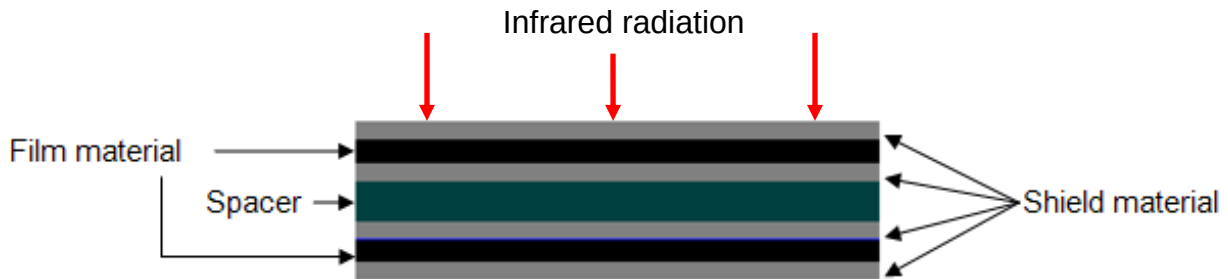


Figure 2: Illustration of MLVSI layers

Certain findings, characteristics and materials used for the components of MLVSI are now discussed.

2.1.1 Shield Material

Cryogenic systems which operate between 300 K and 77 K or lower, make use of multi-layer vacuum super insulation and almost the total heat flux is due to infrared light [8]. The wavelength of infrared light is between 0.7 and 300 micrometers (shorter than those of microwaves but longer than visible light) with a frequency between approximately 1 and 430 THz. Microscopically, infrared light is either absorbed or

emitted when these rays strike an object, increasing the motion of its surface molecules. The amount of infrared light absorbed or emitted is influenced by the temperature of the surroundings (irradiation), the reflectivity as well as the absorptivity and emissivity values of the material's surface finish. Figure 3 illustrates how infrared light is absorbed or emitted through a typical shield material.

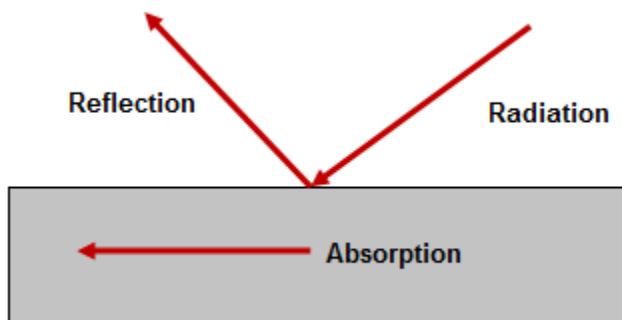


Figure 3: Absorption and reflection processes associated with a non-transparent medium, modified after [9]

Kirchhoff's law states that: at thermal equilibrium, the emissivity of a black body equals its absorptivity. Meaning emissivity is an irradiative property of a surface that provides a measure of how efficiently a surface emits energy relative to a blackbody. In short a poor reflector is a good emitter and vice versa. The symbol ϵ is used for the emissivity of a material and has values in the range $0 \leq \epsilon \leq 1$. The emissivity value of a material strongly depends on its surface finish [9]. A black body has an emissivity value of $\epsilon = 1$, meaning a material cannot thermally radiate more energy than a black body.

According to Incropera *et al.* (2005), absorptivity is a property that determines the fraction of the irradiation absorbed by a surface. The symbol for absorptivity is α , with values in the range $0 \leq \alpha \leq 1$. If the absorptivity is smaller than one then some of the irradiation is reflected. Shield materials used for MLVSI have low emissivity and absorptivity values and radiate most of the infrared light while only absorbing a small percentage of irradiated infrared light.

A typical shield material is a highly polished gold plated polymer film, which has an emissivity of 0.01 and is likely to reflect almost all of the infrared light (Table 1). Highly polished silver and aluminium foil also has promising emissivity values [9].

Table 1: Emissivity of various shield materials [[9]]

Reflector	Emissivity (ϵ)
Aluminium foil	0.02
Copper Highly polished)	0.03
Silver (Polished)	0.02
Gold (Highly polished)	0.01
Stainless Steel (AISI 347)	0.87
Chromium	0.05

2.1.2 Spacer Material

The spacer material is designed to limit heat transfer due to solid conduction between alternative shields. According to Wei *et al.* (2009) spacer material in paper form is most commonly used, whereas the composite paper is replaced with a polymer net, also known as a screen, to further reduce heat transfer. In this way, the contact area through which heat is transferred from one shield to another is much less than with ordinary paper spacers. The tensile strength of the spacer materials has an effect on the installation procedure.

Typical spacer materials used are fibre glass paper, crinkled polyester film and vinyl-coated fibreglass screen although the glass fibre paper has a poor tensile strength. Thus it would take much longer to install glass fibre spacers when compared with glass fabric, which is a stronger material to work with. Spacer weight is of utmost importance when designing cryogenic systems for vehicle applications as the system has to be lightweight. It is believed that fibrous spacers are more promising for cryogenic applications due to the longer heat paths between fibres, although it tends to have lower tensile strength which makes the installation process time consuming [10].

Three main types of fibre matrices are used in spacer material applications and are classified according to tensile strength, heat path and application (Figure 4).

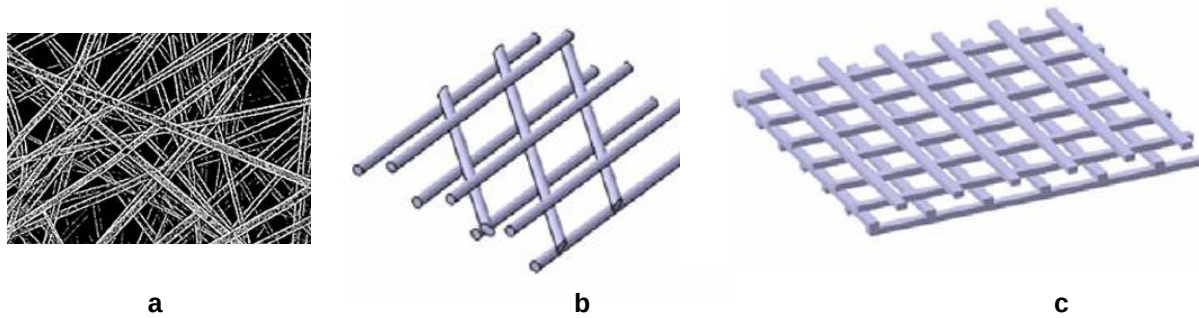


Figure 4: Spacer Matrix Configurations: a) randomly orientated, b) parallel structure c) staggered beam structure [10]

It is believed that the effective thermal conductivity of a fibrous material is dependent on the solid conductivity of the fibrous material, Young's modulus, porosity, imposed pressure and fibre orientation. According to Kwon *et al.* (2009) the longer the heat path of the fibre the more difficult it is for heat to transfer between shields.

The first illustration (Fig. 4a) shows glass fibre paper where the fibres are oriented randomly with respect to a perpendicular main heat flow direction. According to Fricke *et al.* (1990) glass fibre paper with randomly orientated fibres can reach a thermal conductivity of 1.5×10^{-3} W/m·K if the external pressure is held below 1 bar.

The parallel fibre structure (Fig. 4b) illustrates fibres spaced so that a more extended heat path can be generated. This kind of fibre spacing is known to have a lower thermal conductivity than the randomly spaced fibres. Another way to reduce solid conduction is by extending the heat path by the staggered beam method (Fig. 4c). This heat path structure is obtained by placing the rectangular cross-section beams of the first layer at right angles to those of the second layer [11]. The third layer beams are placed half pitch to the first layer ones. The use of rectangular cross-section beams is also beneficial to increasing the beam stiffness. The staggered beam structure will take an I-beam pattern, which contributes to further improvement of tensile strength of the spacer material. Various materials were examined by Kwon *et al.* (2009) for this configuration and they found that polymers exhibit the best performance. It was discovered that the solid conductivity was also dependent on the angle of the adjacent beams.

2.1.3 Film Material

Film material is one of the most important components in layered insulation because it adds strength to the shield material in order to be able to wind it around a cryogenic tank. The number of film layers wound in a multi-layer application is called the *layer density* of the MLVSI. Alternating layers of these film material, shields and low conductivity spacer material, is called an *insulation blanket*.

According to Lebrun *et al.* (1992) typical film materials are Mylar, Kapton, Tedlar and Teflon. A commonly used film and spacer material is Mylar with a vacuum deposited aluminium coating on one or both sides of the polymer sheet and a fibreglass paper spacer. This type of layered insulation represents the benchmark for comparison [12]. Mylar can be produced in sheets as thin as 3.8×10^{-4} m and has a thermal conductivity of 0.151 W/m·K whereas Teflon sheets have a thickness of 12.7×10^{-4} m and a thermal conductivity of 0.209 W/m·K. Mylar also has the lowest density and thermal expansion of these two materials.

2.2 Common Behavior of MLVSI

According to Wikstrom (1999) MLVSI is highly anisotropic and is very sensitive to compressive loads. When multi-layer insulation is installed too tight the layers compresses and the thermal conductivity increases due to a decrease in gap distance between alternative layers. If the multi-layer insulation is too loose, thermal conductivities will be influenced on the bottom side of the tank that is being insulated. Multi-layer insulation requires careful attention during installation, and according to John (2009) it is difficult or impossible to maintain the gap distance between the film layers [13] [14].

Thermal behaviour of multi-layer insulation blankets (MLIB) was measured by Benda *et al.* (2000). The measurements focussed on large MLI samples for use in industrial plants. According to Benda *et al.* (2000) the research was done with the aim to optimize MLI and their thermal behaviour. Two testing cryostats were used in measuring the thermal performance of MLI [8]. The first was a vertical cylinder cryostat surrounded by

thermal insulation in a vacuum. A cold boundary temperature was generated inside the cylinder making use of liquid helium at 4.2K. The warm boundary was gaseous nitrogen at 78K. The second test apparatus was a flat plate configuration with cold and warm boundary temperatures. Accordingly, this eliminates parameters like material emissivity and the view factor. Film materials, shield material as well as spacer materials used in cryogenic insulation blankets were tested. Results showed that the best combination for a cryogenic blanket is double aluminized Mylar with a crinkled aluminium shield and a fibre glass paper spacer. The overlap closing method used was aluminium tape or Velcro which was used in more effective installation to join edges, and where overlapping posed a problem. This resulted in more heat transfer, due to the stitching and extra material added to the blanket. The problem was overcome by using a strip of Mylar film between alternating Velcro layers to limit heat transfer.

The number of spacers varied from layer to layer: cold side, 3 spacers between 2 reflectors, warm side, 1 spacer between reflectors, for a total of 15 spacers. This specific blanket generated the best low thermal conductivities at vacuum pressures below 10^{-5} Pa. The reason for using crinkled aluminium shield is that only certain points touches the spacer material, which leads to less heat transfer.

Lebrun *et.al.* (1992) worked on another project where thermal insulation was required for the CERN Hadron Collider to operate as an effective thermal barrier under any change in vacuum conditions. According to Lubren *et.al.* (1992) a promising combination for MLVSI was a double aluminised polyester film with a polyester net spacer material.

The performance of commercially available MLI was tested by S.D Augustynowicz and J.E Fesmire (2005). Combinations of aluminium foil, fibreglass paper, polyester fabric, silica aerogel composite blanket, fumed silica, silica aerogel as well as other novel materials were tested with their results presented in Table 2.

Table 2: Thermal conductivities of typical MLI sample configurations of materials tested at 13mbar, modified after [12]

Description of Insulation	Vacuum (mbar)	Conductivity (mW/m·K)
Layered composite insulation with fiberglass paper and fumed silica dispersion	13	6.07
MLI (aluminum foil and fiberglass paper spacer), 40 layers at 1.8 layers/mm	13	13.6
Layered composite insulation with polyester fabric and fumed silica dispersion	13	9.66
Layered composite insulation with fiberglass paper and fumed silica dispersion	13	7.71
Layered composite insulation with fiberglass paper and fumed silica dispersion	13	6.82
Layered composite insulation with polyester fabric and fumed silica dispersion	13	8.78

The insulation samples (Table 2) were tested at warm and cold boundary temperatures of 90 K and 300 K respectively. It was concluded by these authors that aluminium foil with no film material can be used with glass fibre paper spacers. Multi-layer insulation blanket with a layer density of 40, at a vacuum of 13 mbar without any film material, resulted in thermal conductivities as low as $13.6 \text{ mW/m} \cdot \text{K}$. A MLI (Aluminium foil and fibreglass paper spacer.) blanket tested by Fesmire *et.al* consisting of 50 layers of aluminium foil and glass fibre paper showed a remarkable low thermal conductivity of $0.06 \text{ mW/m} \cdot \text{K}$ at a vacuum pressure of $4 \times 10^{-5} \text{ torr}$.

Further improvements were made to MLI by perforating the shield material. The thermal performance of Perforated Multi-Layer Insulation Blankets (PMLIB) was experimentally tested [15]. The cold boundary temperature of a calorimeter was insulated with layer densities of 20, 30, 40 and 50 layers respectively. It was concluded by Wei *et.al.* (2009) that the thermal performance of PMLIB was highly affected by the structure and shape. An insulation blanket with a layer density of 50 allowed a heat flux of 0.77 W/m^2 .

2.3 Testing Methods for MLVSI

It is believed that testing the insulation effectiveness of layered insulation is a complicated process because of the extreme care that must be taken with fabrication and installation. Most of the apparatus used for testing MLVSI at these low boundary temperatures make use of the cryogen boil-off calorimeter method. A cold mass like liquid nitrogen is poured into an insulated container and weighed. As heat is being transferred through the insulation material, the liquid starts to warm up and evaporates due to its low boiling temperature. The liquid mass evaporated is directly proportional to the amount of heat being transferred through the insulation to the cryogenic fluid.

Bapat *et al.* (1990) believes that a cylindrical apparatus operating on the boil-off measurement principle is the most preferable for testing the effectiveness of a MLVSI configuration composite. According to Kagner *et al.* (1969) the cylindrical method has advantages like maximum surface area and minimum edge effect area [16]. Using this method it is easy to control secondary heat leaks but these cylindrical arrangements cannot be used to test the influences of mechanical loads between alternating layers.

An improved method for testing rolled insulation material was developed at the John. F Kennedy Space Centre (Fig. 5) [17]. In this method a stainless steel cylinder is filled with liquid nitrogen to form the cryostat. The multi-layer insulation is wound onto a copper sleeve to a certain layer density by using a wrapping machine (Appendix A) and then slid over the stainless steel cylinder. Thermal performance of MLVSI is tested by taking temperature readings between alternative insulation layers. This boil-off calorimeter system enables direct measurement of the thermal conductivity of the insulation material. To prevent heat gained through the ends of the cryostat NASA has made use of thermal guards consisting of liquid nitrogen containers at the ends, and only the performance of the insulation around the liquid nitrogen chamber is tested.

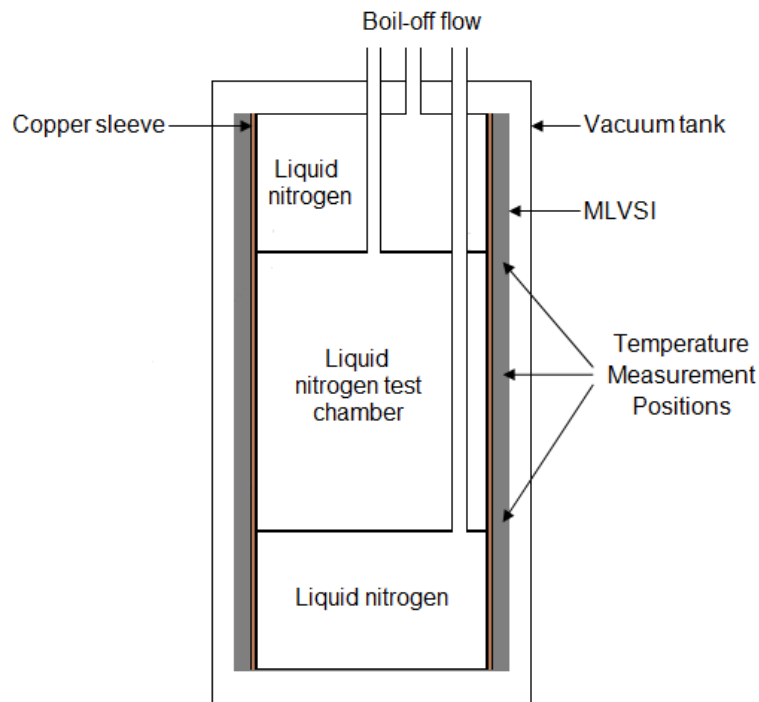


Figure 5: Illustration of liquid nitrogen calorimeter for testing the effectiveness of MLVSI, modified after [17]

The problem with this system is that it required a liquid nitrogen supply that added additional heat to the system by conduction through inlet pipes [17]. Fesmire *et al.* (2008) however, carried out multiple tests on various composite insulation materials using this method and it was found that the predicted results were consistent with their experimental results.

NASA has designed another test apparatus at the Cryogenics Test Laboratory for the measuring of MLVSI performances (Fig. 6). In this apparatus the setup consists of a vacuum chamber with a stainless steel cylinder filled with liquid nitrogen.

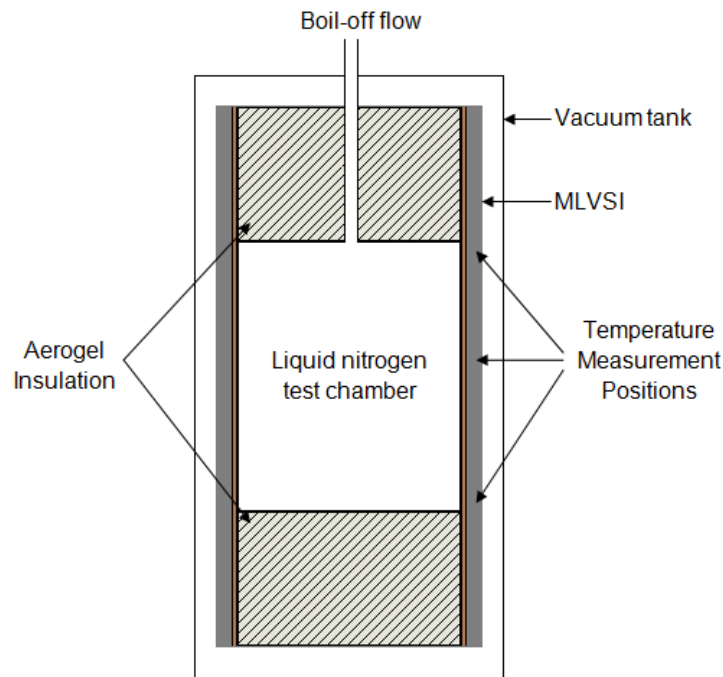


Figure 6: Illustration of a boil-off calorimeter for testing MLVSI, modified after [17]

The inner assembly is easily removed and can also be placed in a wrapping machine for installing MLI. Heat through the ends of the inner cylinder is minimized by thermal guard discs made of aerogel with silver plated film material in between. The boil-off weight is measured from which the insulation performance is calculated. This test method provides more consistent measurements due to less heat transfer through inlet piping.

2.4 MLVSI Installation

Shield and spacer material is normally supplied in rolls for purposes of installing onto cryogenic containers. In the case of a cylindrical shape container, like the two test methods discussed above, the insulation is wound around the container.

NASA used a wrapping machine (Appendix A) that consisted of three rollers which were used for different multi-layer insulation test configurations. The cylinder to be filled with a cryogenic fluid is placed on the machine and then a set of rollers applies MLI to the container by rotating of the rollers. An improved method and apparatus was invented

by Gonczy *et al.* (1988) to fabricate multi-layer blankets. According to Gonczy *et al.* (1988) this machine is able to wind multiple layers around a mandrel and to cut the material along a line during winding or on completion of the winding procedure. The blanket is removed after the edges are bound together along the circumference of the mandrel. Specific layer densities can be achieved when the tension on the two materials is adjusted [18].

2.5 Summary

The thermal behaviour of layered insulation blankets is not only a function of physical properties such as thermal conductivity, emissivity and absorptivity but also of the type of installation, compressive forces between layers as well as the joining method of the inner and outer tanks.

The problem faced with the discussed MLVSI testing apparatus is that it only simulates MLVSI performance around a cylinder and not the performance if installed around a typical pressure vessel with more complicated geometries.

Chapter 3: Experimental Design

3 Introduction

To determine the effectiveness and degree of edge effects of application of MLVSI to a typical cylindrical pressure vessel (for storage of liquid hydrogen or any other cryogenic fluid) and to test fluid inlet/outlet coupling configurations an experimental cryogenic system is required. It was thought that the experimental system based on the boil-off calorimeter principle discussed in Section 2.3, would provide the base for such a design.

Although this work is aimed at determining the effectiveness of application of MLVSI to a typical pressure vessel containing liquid hydrogen, liquid nitrogen needs to be used as the cryogenic fluid since the facilities to safely work with hydrogen is not available. Although the thermal conductivity or heat flux into a cryogenic system is a function of temperature (with liquid hydrogen at a lower storage temperature than liquid nitrogen) it is thought that the results obtained would further guide MLVSI application to such hydrogen containing cryogenic systems and their design – especially for vehicular application. The following aspects needed to be addressed and are discussed in this chapter:

- Boil-off calorimeter method modifications
- Operational requirements
- Material and component requirements

3.1 Experimental Cryogenic System

The boil-off calorimeter method works on the evaporation of a liquid principle. An insulated container is filled with a cryogenic fluid such as liquid nitrogen and as heat is transferred through the insulation the liquid nitrogen (or for that matter liquid hydrogen) warms up and evaporates. The amount of evaporated nitrogen gives an indication of how much energy (heat) is required to evaporate the amount of liquid nitrogen. A largely evaporated amount of liquid nitrogen indicates poor insulation performance and vice

versa. According to Bapat *et al.* (1990) this is a simple, but accurate method for measuring thermal insulation properties at very low temperatures.

A schematic of the required experimental cryogenic system, based on the boil-off calorimeter method, is shown in Figure 7. The experimental system needs to consist of an inner and outer tank to provide for essential vacuum insulation.

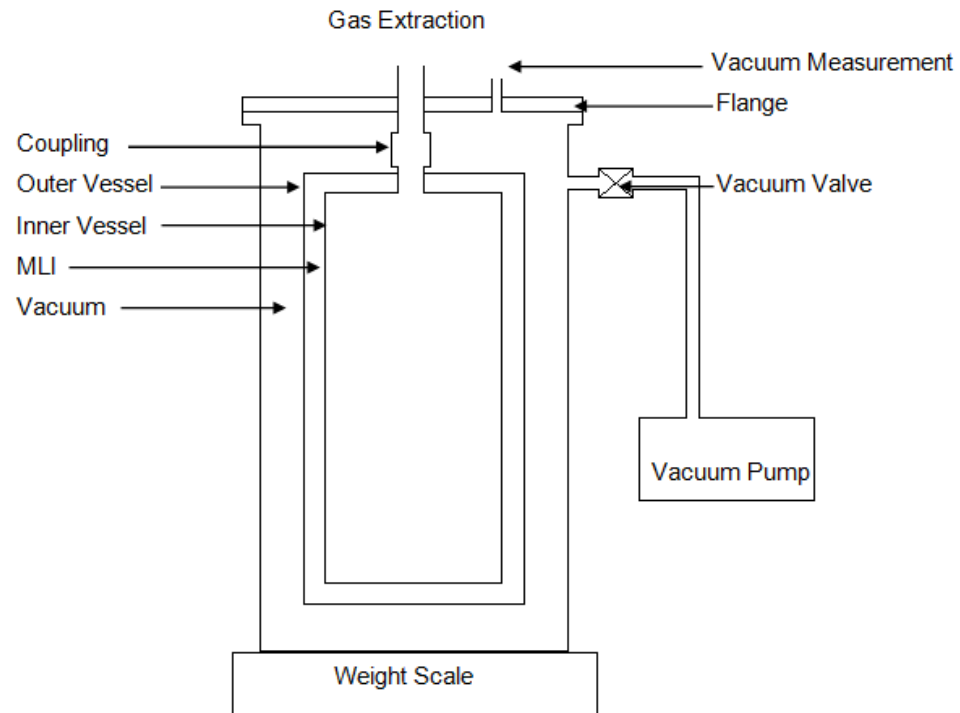


Figure 7: Schematic illustration of the required experimental cryogenic system

The inner tank is to be mounted to the outer tank by making use of a coupling that can also act as an inlet/outlet nozzle. A vacuum supply system is to be connected to the outer tank and the outer tank should also provide for protrusions and fittings for vacuum and temperature instrumentation. The system needs to be provided with a convenient top opening configuration so that the inner tank with insulation can be removed after tests to easily change assembly configurations and/or insulation layers.

3.2 Operational Requirements

The function of the test system is to determine the thermal performance of certain insulation and coupling configurations by measuring nitrogen boil-off. This means that the inner vessel should hold enough liquid nitrogen for effective testing and that material weight should be kept to a minimum as the apparatus needs to be weighed on a high precision bench scale to determine boil-off. For testing to be accurate and safe, the experimental location should be well ventilated and at constant ambient temperature and pressure. A vacuum system and gate valve needs to be selected as the vacuum pipeline is required to be closed off after vacuum pumping.

Testing should be done in the shortest time period possible, which means the inner vessel must be easily separated from the outer vessel to ensure fast insulation replacement. The coupling has to carry the total weight of the inner tank to prevent it from failing under tensile loads; however it should also limit heat transfer between the cryogenic fluid and the outer tank.

Liquid nitrogen at a cryogenically low temperature can embrittle structural materials and they can also undergo a ductile-brittle transformation. Because of this characteristic attention had to be given to the selection of materials. Material which comes into contact with moist can form ice causing the pipes and fittings to be plugged which can result in an explosion. The pipes and fittings have to be selected so that it does not fail under these cryogenic conditions.

High vacuum levels require a vessel with acceptable hoop strength and components to ensure integrity while testing proceeds. Due to system vacuum requirements, the pipeline components should prevent particles from being sucked in by the vacuum pump and should ensure efficient sealing properties. For sealing purposes, it is necessary that all the pipeline components have a very fine surface finish to be able to seal properly. Also, they should not corrode as this will form metal particles which can damage the vacuum pump vanes.

Nitrogen gas is harmful when inhaled and gas extraction fans are required to vent the gas to an unoccupied space. A stand is required to add stability to the system, ensuring practical and safe system installations and analysis. The stand needs to be modifiable so that it can be used for various practical experimental purposes. It should keep the extraction fan in place while testing and also set the height of the extraction fan. Between tests the extraction fan needs to be removed, whilst the stand is used to hold the upper dome in place to mount thermocouples to the coupling. The stand should also be able to hold the inner tank in place in order to install the insulation layer by layer while also avoiding compression

Chapter 4: Detail Design

4 Background

This part of the report describes the mechanical design of the experimental cryogenic system and its components for testing MLVSI and coupling nozzle configurations applied to a spherical shaped pressure vessel as based on the requirements presented in Chapter 3. Section 4.1 and Section 4.2 address the following aspects of the design:

- Cryostat externals
- Cryostat internals

The components of the final design are illustrated in Figure 8.

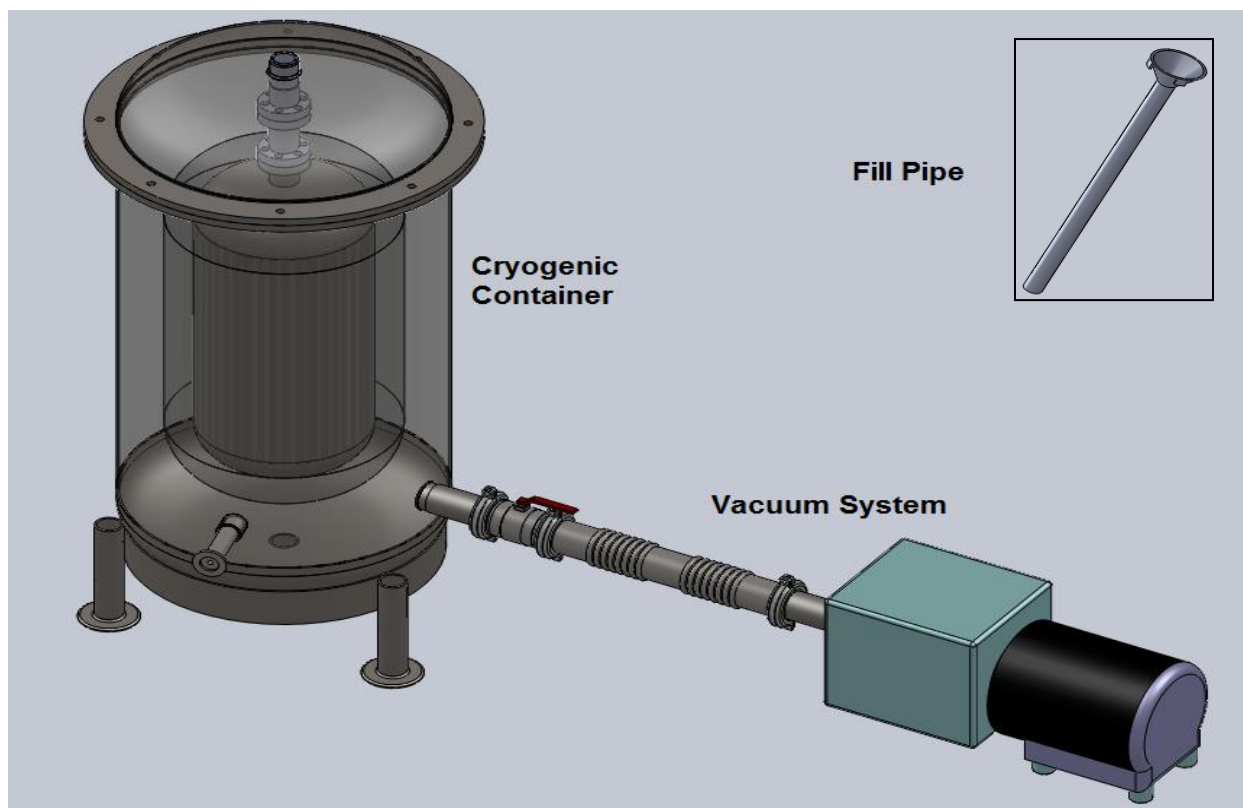


Figure 8: Transparent CAD model of the experimental system

4.1 Cryostat Externals

The cryostat externals consist of the:

- Cryostat outer tank and the
- Vacuum pipeline

4.1.1 Cryostat Outer Tank

The outer tank is a 50 litre, 11 bar pressure vessel with an outside diameter of 285mm and a wall thickness of 2mm, obtained off-the-shelf. The experimental cryogenic system operates at a pressure difference of 1 bar and no calculations were required regarding safe outer tank stresses because of the rated vessel obtained. The tank operates at a higher temperature than the inner tank although the neck area, near to the inlet coupling, can reach temperatures as low as 240K. This makes it possible to manufacture the outer tank and flanges from plain carbon steel as the brittle transition temperature of plain carbon steels is in the range of 225 K which is lower than the expected operational temperature.

The flange and sensor protrusions are manufactured from plain carbon steel as it is welded to the outer tank. Flanges are coupled using a 5 mm vinyl O-ring to create an air tight seal by using 8x6 mm stainless steel bolts. Figure 9 illustrates a modelled cross section of the assembled gland flanges design.

O-rings were selected because they are relatively inexpensive and requires lower seating stresses than flat gaskets. Note was taken of the direction in which pressure is applied. The internal diameter dimensions of the groove were designed to fit the internal diameter of the O-ring for pressure applied from the outside since a vacuum is generated. For a 5mm O-ring the width and height of the groove was calculated to be 6.5mm and 4mm respectively. For the groove height a tolerance of $\pm 0.05\text{mm}$ was allowed.

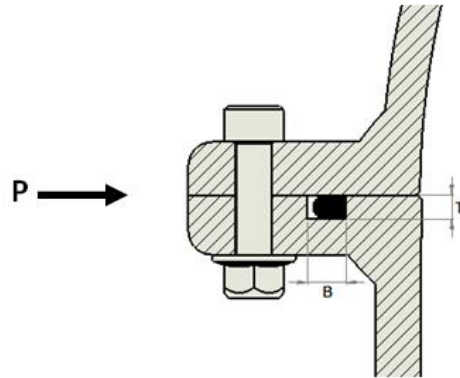


Figure 9: Schematic of the cross section view of the O-ring gland flanges

A threaded flange welded to the outer tank dome is provided for, in order to mount the inner tank supply coupling to the outer tank dome. An exploded view of the model of the outer tank is shown in Figure 10.

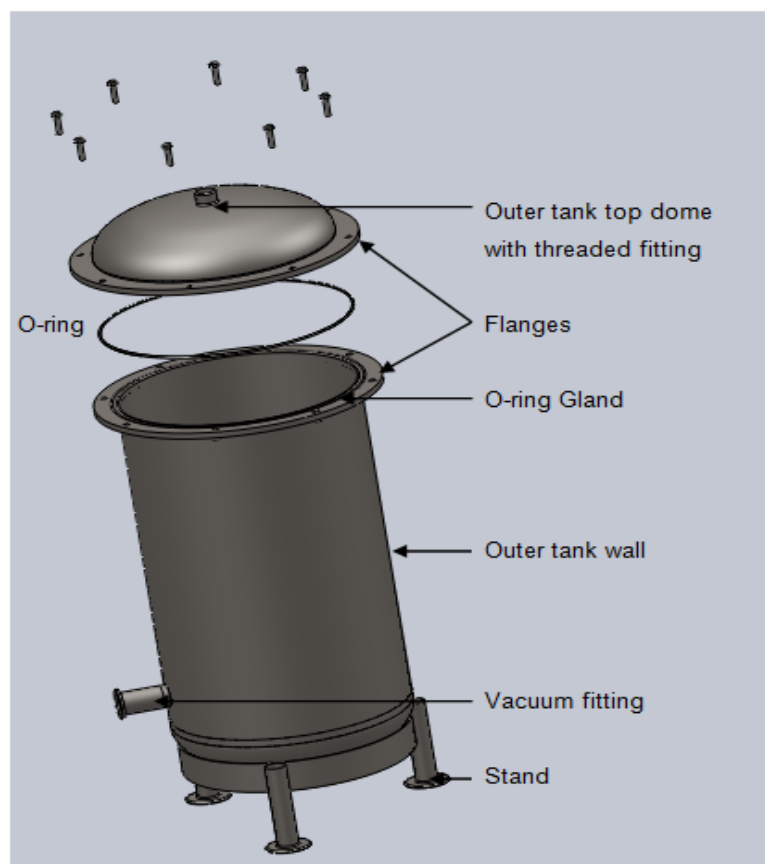


Figure 10: CAD model exploded view of the cryogenic system outer tank

4.1.2 Vacuum Pipeline

An exploded view of the vacuum pipeline is shown in Figure 11 and consists of components that are obtainable off-the-shelf.

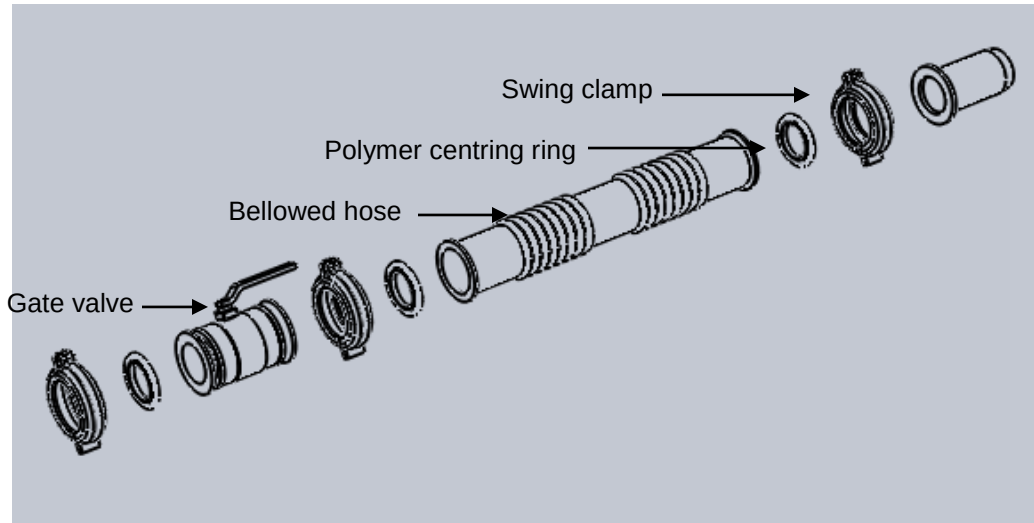


Figure 11: Exploded view of the CAD model of the vacuum pipeline

For weighing purposes, the vacuum pipeline can be disconnected from the system. If not the weight and stiffness of this pipeline will have an influence on scale measurements. After weight measurements are taken the pipeline is connected again to generate vacuum if leaks are a problem during testing. To be able to disconnect the pipeline after sufficient vacuum levels have been reached a vacuum gate valve with the smallest leakage rate possible, is provided for. This gate valve is connected to the outer tank vacuum flange protrusion using the aluminium swing clamp and Viton O-ring seal such as illustrated in Figure 11. A bellowed hose is installed between the pump and the gate valve to prevent it from damaging the scale by vibrations transferred from the vacuum pump in operation. Vacuum grease is added to the sealing surface of each component to ensure optimum sealing.

4.2 Cryostat Internals

The cryostat internals consist of the:

- Cryostat inner tank
- Inlet nozzle coupling

4.2.1 Cryostat Inner Tank

A CAD model of the designed system internals is shown in Figure 12. The design (Design and manufacturing drawings presented, Appendix F) provided for a cylindrical gap between the inner and outer tanks of 60mm to be able to install enough MLVSI to the inner tank and still have a reasonable vacuum space between the outer tank wall and the MLVSI.

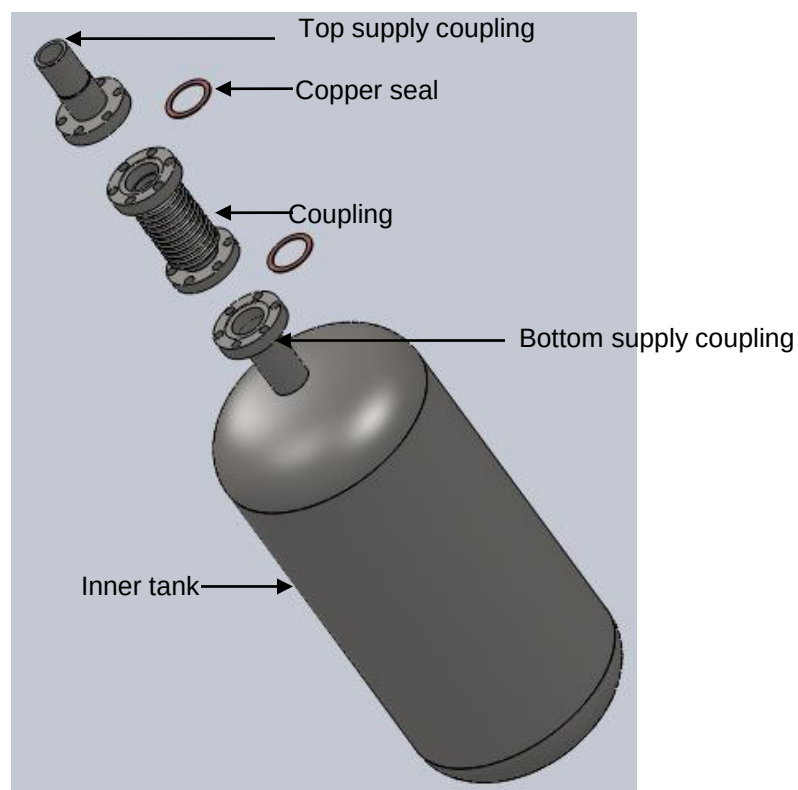


Figure 12: Exploded view of the CAD model of the experimental system internal components

With an outer tank inner diameter and length of 280mm and 570mm respectively, the inner tank diameter is calculated to be 155mm with a length of 330mm. The inner tank

needs to be 80% filled during testing in order to prevent liquid nitrogen being pushed out during gas evaporation. The amount of liquid nitrogen used per test conducted is calculated to be 3.5 litres. The inner tank is operating at 77 K and materials such as ferritic stainless steel and polymers have been identified as non favorable materials due to its ductile to brittle transition at this low temperature. Polymers cannot handle these temperatures either and were therefore not employed. Materials considered for the inner tank is shown in Table 3.

Table 3: Considered materials to manufacture cryogenic inner vessels

Aluminum 2014-T6	Ti-6Al-4V	70Cu-30Zn	Stainless steel 316
Aluminum 5052-O	Inconel 718	Composites	Stainless steel 304

According to Fesmire *et al.* (2005) heavy wall stainless steel construction provides maximum thermal stability and minimum temperature gradients for such tanks. Austenitic stainless steels are useful at cryogenic temperatures and are also used in plants handling liquefied gas. SAE 316 and SAE 304 were considered for this design due to their unique combination of properties. Based on relative cost, 2mm stainless steel SAE 304 sheet was selected for the inner tank manufacturing. The maximum operating pressure is 1bar with the result that the tank cannot be seen as a pressure vessel (ASME standards, Division 1 of Section 8 for pressure vessels operating above 103.4 kPa).

A bottom supply flange made from stainless steel was provided and welded to the inner tank. With the top and bottom supply flanges it should be possible to change and improve coupling designs without removing some system components.

4.2.2 Inlet Coupling/Nozzle

The inner tank inlet coupling is a critical component in the experimental cryogenic system and was designed to limit conduction through the neck area of the cryostat. When making use of cryogenic systems it is thought that most of the transferred heat to

a cryogenic liquid is due to radiation from the outer tank wall and conduction through the inner coupling. To ensure limited heat transfer, a coupling was designed to replace the conventional straight inlet pipe also employed in the experimental work.

According to Fourier's law, the heat flux through an object is given by.

$$q = k \frac{\Delta T}{L}$$

where

k	-	Material's thermal conductivity,
L	-	Heat transfer path and
ΔT	-	Temperature difference

The heat flux is given in W/m^2 which means that the heat flux has to be multiplied by the heat transfer area to calculate the total transferred heat. Thus, the heat transfer is influenced by the length of the inlet coupling, through which heat has to be transferred, as well as the cross sectional area. This acted as a starting point for coupling design. With the above taken into consideration it was decided to design the coupling in the form of a bellow. With the specific geometry of a bellow it is possible to minimize the gross sectional area but still have a strong enough coupling under high vacuum to avoid failure due to implosion as vacuum is generated. The heat path through a bellow is illustrated in Figure 13.

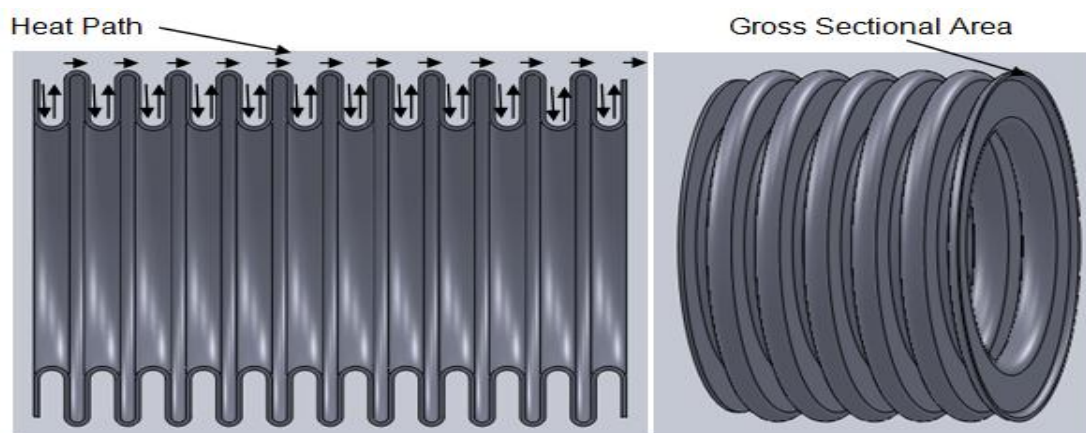


Figure 13: CAD model illustrating the heat path through a thin walled bellow

Due to the specific form of the bellow, a longer heat path is generated than with an ordinary inlet pipe. In order to mount the bellow between the inner and outer tank, it was decided to make use of two flanges welded to both ends of the bellow. Such a design would enable easy fitment and dismantling of the coupling during configuration changes.

Chapter 5: Manufacturing and Assembly

5 Introduction

The majority of components used for the experimental cryogenic system were manufactured by the author. Assembly was carried out in the Mechanical Engineering workshop. Manufacturing drawings are presented in Appendix F.

5.1 Inner Tank and Coupling Manufacturing

The inner tank consists of a rolled sheet metal cylinder welded to two self-manufactured domes. In order to produce these domes a male and female die was designed and produced. The cutting of the dies on a CNC milling machine, for the production of the header domes, is shown in Figure 14. These dies were manufactured from mild steel as only a few domes needed to be produced.

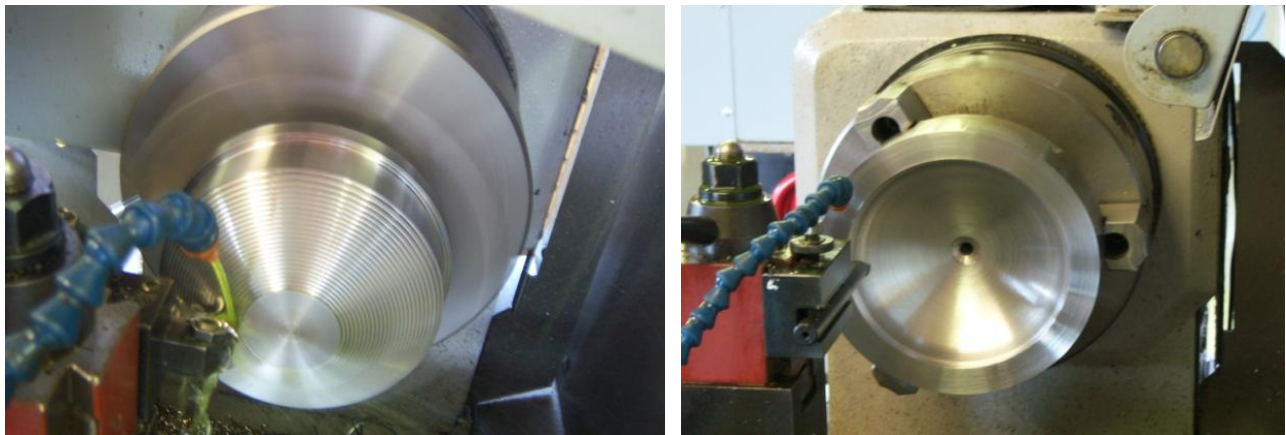


Figure 14: Inner tank dome male and female die manufacturing

In order to remove the dome from the die after forming and to ensure the dome radius was correctly manufactured, compensation was made for material elastic springback by allowing for clearance between the male and female dies to be about the billet thickness. In most cases, the clearance between a male and female die is calculated as the billet thickness plus twenty percent of this thickness. The clearance between alternative dies for a 2mm billet was therefore calculated to be 2.4mm. Figure 15

demonstrates the final products obtained after machining. The centre hole in the female die facilitated the removal of the product after pressing was carried out.



Figure 15: Inner Tank Male and Female Dies

Accurate work was of utmost importance to ensure good material flow while pressing continued. A 900kN press was used and the hydraulic force to produce a single dome was calculated to be approximately 9 tons. Figure 16 shows the beginning and end of the pressing process.



Figure 16: Inner tank dome pressing process

It was important to centre the male die as well as the billet before the pressing started in order to ensure a perfectly symmetric shaped dome. Figure 17 illustrates a dome that was manufactured from a 200mm diameter billet with a thickness of 2mm.

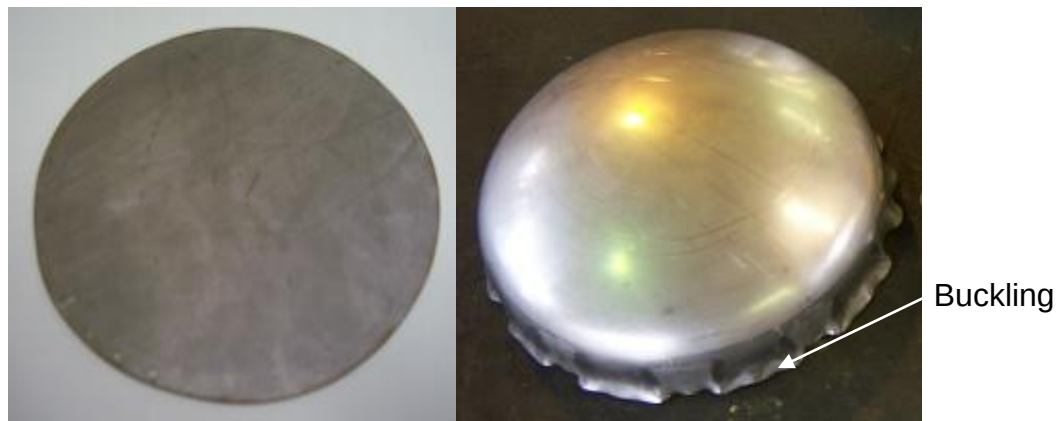


Figure 17: Manufactured dome indicating buckling

The dome was buckled at the outer radius and it was established that the female die was too shallow and that the material deformed before the final radius bend was generated. It was essential that the final radius bend should be parallel to the cylinder wall. The dies were redesigned and machined deeper in order to achieve the final radius bend. A cross sectional schematic view of the re-machined female die is shown in Figure 18.

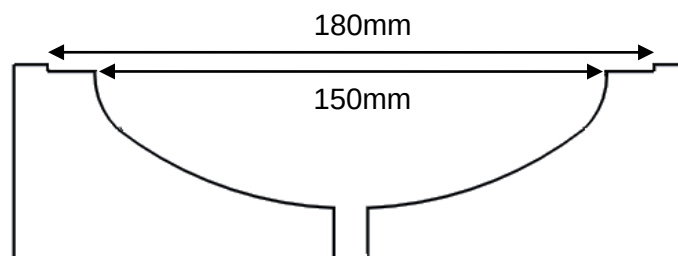


Figure 18: Cross sectional schematic view of the redesigned female die

A fillet was machined in the female die to hold a 180mm diameter billet in place when pressed. This diameter billet was chosen because no excess material was present to give an unshaped finish. Various billet sizes were produced and formed with and without the use of grease as lubricant. The results of such produced dome sizes is illustrated in Figure 19.



Figure 19: Manufactured 150mm Ø domes from various billet sizes

Although both the 230mm and the 180mm diameter billets produced with grease, provided promising results, the 180mm diameter billet was preferred as the extra material on the edges of the 230mm diameter billet had to be removed before it could be welded to the inner tank wall. It was also found that with the use of grease the production process provided a smooth surface finish. Tungsten inert gas (TIG) welding was used to weld the domes to the cylinder to produce the vessel.

The assembled inner tank on a stand, that was also manufactured, is shown in Figure 20.

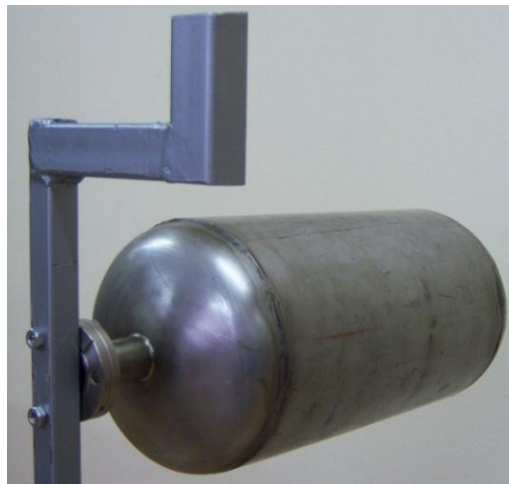


Figure 20: Illustration of the cryogenic inner tank mounted to a stand to facilitate MLSVI installation.

The bellow with the calculated dimensions could be obtained off-the-shelf and was welded to machined flanges. A photo of the manufactured coupling is shown in Figure 21.



Figure 21: Photo of the assembled and weld-joined inner coupling provided with flanges

5.2 Application of MLVSI

It was decided to use MLVSI that consists of aluminum foil shield materials and glass paper spacers with randomly spaced fibers. These materials have a combined layer thickness of 1.8 layers/mm and a thermal conductivity of 13.6 mW/m·K at 13mbar. It was provided in sheets by Cryoshield (Pty) Ltd and had to be cut before application.

In order to reduce heat transfer due to edge effects when installing MLVSI on the pressure vessel with spherical headers, special attention was given to the application process of the acquired insulation. It was required to cut the insulation material layer by layer when installing it to the inner tank. The aluminium foil and spacer material (one layer) had a combined thickness of less than one millimetre with very low tensile stress properties. This complicated process of cutting was very time consuming and a jig was manufactured out of Maysonite to cut the insulation material. The cutting process using the jig is illustrated in Figure 22.



Figure 22: Illustration of MLVSI cutting using a jig over-lay on the insulation with a newspaper page as intermediate layer

The aluminium foil together with the paper spacer was placed face down on a table's surface. Due to the small layer thickness and low tensile strength of the insulation the aluminium foil piled up when cut. This was prevented by placing paper or newspaper over the aluminium foil and by cutting from the inside outwards along the template edges which prevented the insulation layer from tearing. For limitation of radiation it was

critical that the reflective side of the aluminium foil faced up to the outside of the tank. Figure 23 illustrates the cut configuration of one layer of insulation after the cutting template and pile-up precaution paper layer, was removed.

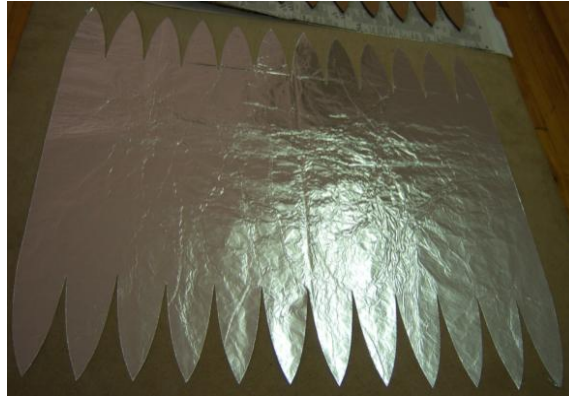


Figure 23: A single layer of MLVSI ready to be installed to the inner tank

In order to insulate the inner tank alternative layers were folded over the dome ends of the inner tank (Fig. 24). Layers were placed one over the other to cover the entire surface area. The folding was managed in such way to eliminate possible edge effects and the second layer was rotated 30° with respect to the previous one to overlap parting lines of the covered layer. The gap distance between overlapping layers influences thermal conductivities of a MLVSI assembly; therefore, the stand was used to hold the inner tank in place while applying MLVSI to assist with the folding technique (Fig. 24).

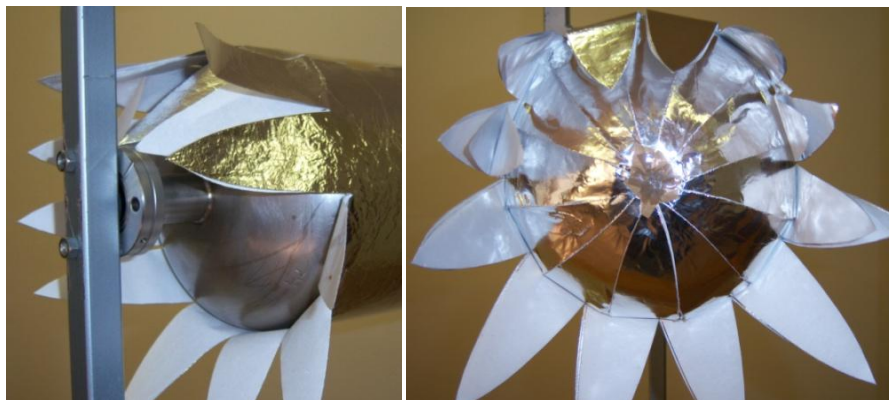


Figure 24: Illustration of the folding process in order to limit edge effects

Chapter 6: Experimental Setup and Procedure

6 Experimental Setup

The experimental setup to test the effectiveness of the MLVSI application and the coupling configurations is illustrated in Figure 25.

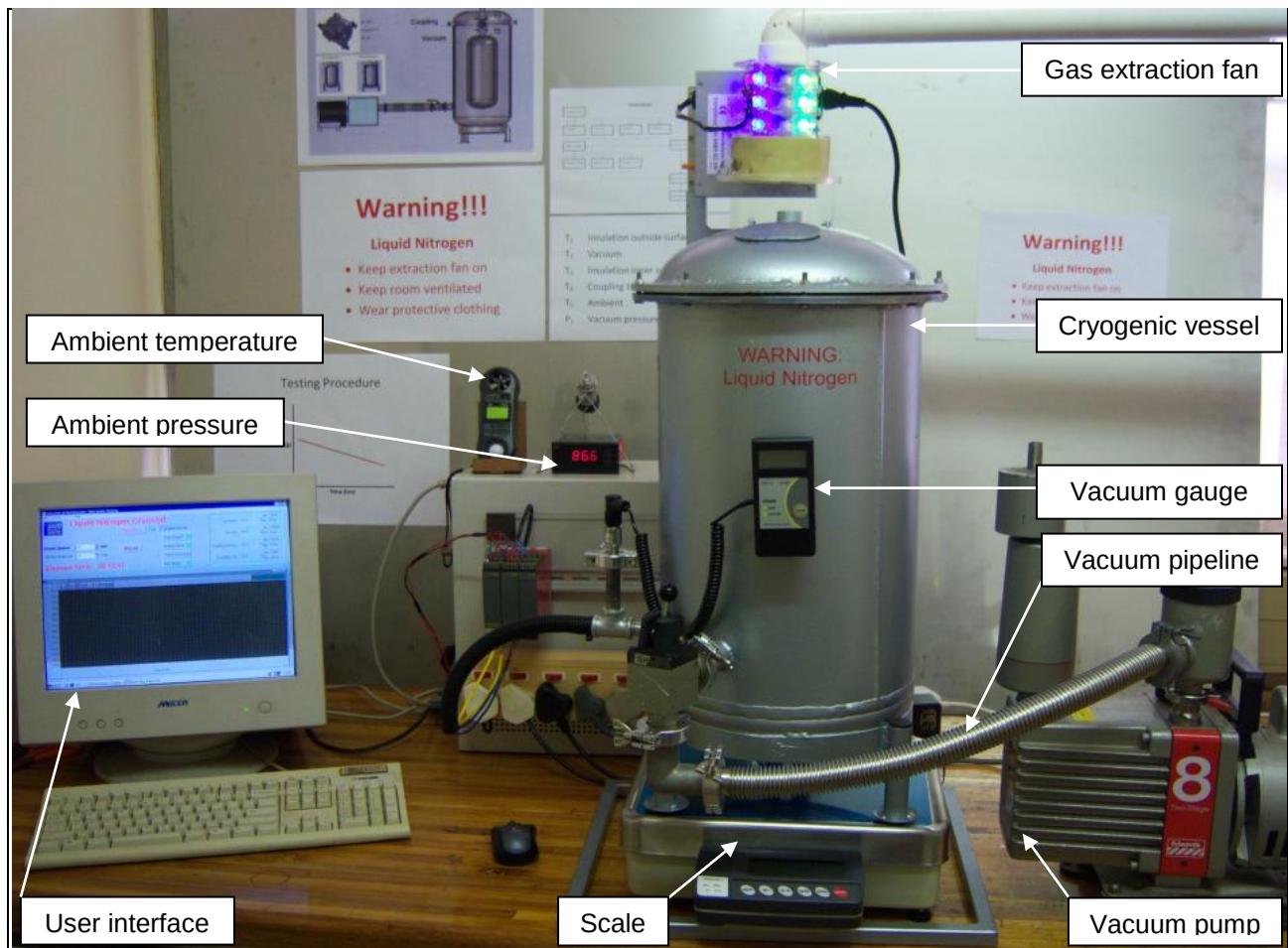


Figure 25: Photo of the experimental cryogenic system for measuring the performance of MLVSI around a vessel with spherical shaped end caps

It consists of three sub-assemblies: The vacuum supply, the cryogenic vessel and the measuring instrumentation. The vacuum supply consisted of a pipeline connected to a vacuum pump in order to generate sufficient vacuum in the insulation space.

Vacuum pumps are widely available in different sizes and designs. A two stage rotary vane vacuum pump (Edwards E2M18 ATEX) was selected, based on the low vacuum pressures that could be reached with this vacuum pump. The E2M18 ATEX rotary vane pump used has an overload device with air tight pumping chambers and gas ballast control. These functions enable the pump to reach vacuum up to 10^{-3} torr. The performance chart of two models namely the E2M18 and E1M18 vacuum pumps respectively with and without gas ballast, is illustrated in Figure 26.

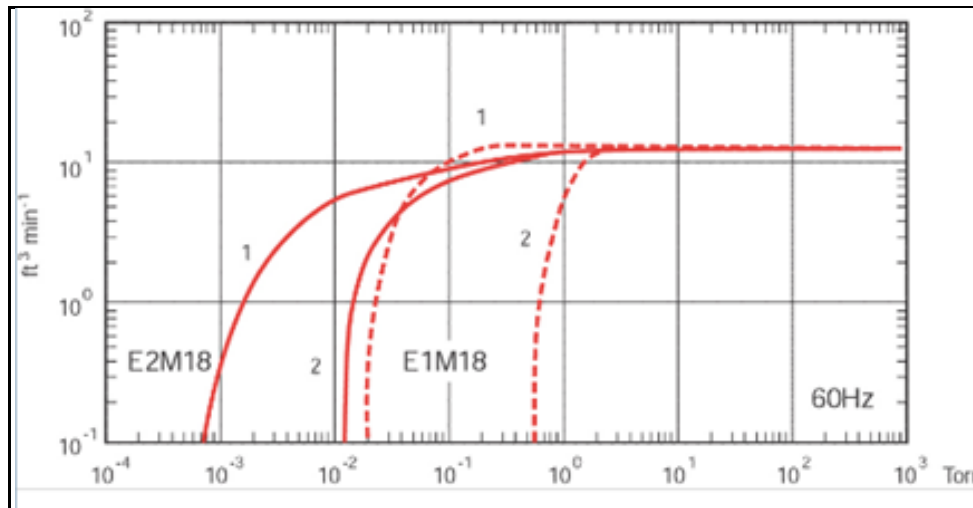


Figure 26: E2M18 and E1M18 vacuum pumps performance chart

The vacuum pumping rate indicates (Fig. 26) how fast a certain volume container can reach a vacuum and is given in cubic feet per minute (ft^3/min). Due to the small volume in the insulation space between the two tanks, it was calculated using this chart that the required vacuum could be reached in just a few minutes. The solid line indicates the pump's performance with gas ballast.

Figure 27 illustrates the results obtained from experimental tests carried out on pumping speed vs. vacuum pressure for the E2M18 model. The results show that a vacuum level of 10^{-4} torr could be obtained by this model. Thus practically this pump had the capacity to generate higher vacuum levels than theoretically required. With the experimental setup having an insulation space of 0.015m^3 it would take less than a minute to generate a vacuum of 10^{-3} torr by making use of this pump.

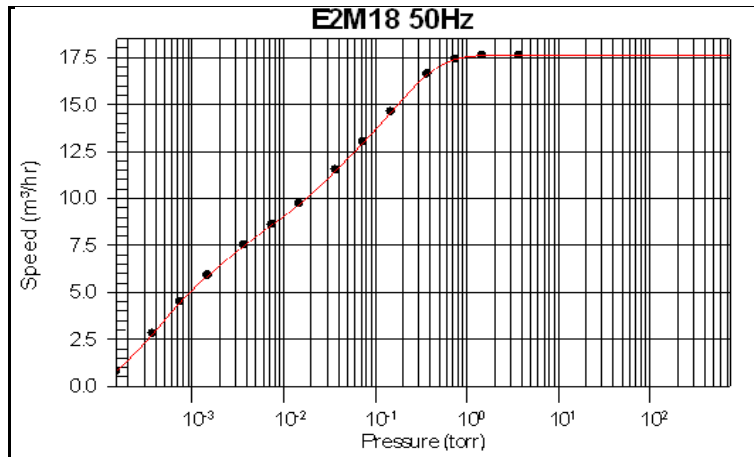


Figure 27: E2M18 vacuum pump experimental performance chart

Vacuum and temperature readings were logged throughout testing and therefore it was important to use accurate and reliable measuring instruments. It was decided to measure vacuum with a piezo-resistive ceramic sensor. This sensor can measure vacuum levels from 0.001mbar to 100mbar and was connected to the tank using a c-clamp with polymer seal. The instrument functions in such way, that when it is under the influence of pressure a thin diaphragm is bent with a resistor bridge on its back. The bending force causes the measurement bridge to come out of tune, which creates the measurement reading, for the applied pressure. The instrument makes use of a 9 volt battery which makes it possible to attach it to the tank without coupling to an external supply. The calibrated vacuum sensor is shown in Figure 28.

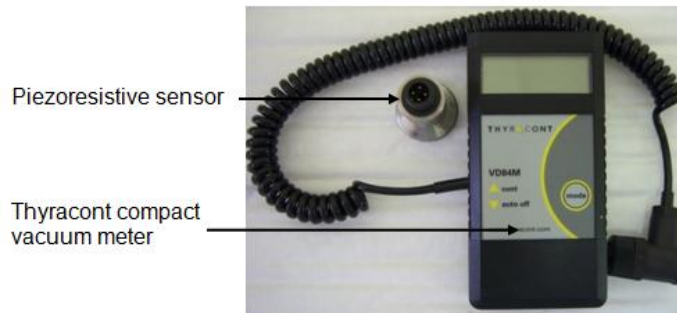


Figure 28: Thyracont high vacuum sensor and control unit

Various temperature readings were taken mostly inside the cryogenic vessel. Temperature measurement positions are shown in Figure 29.

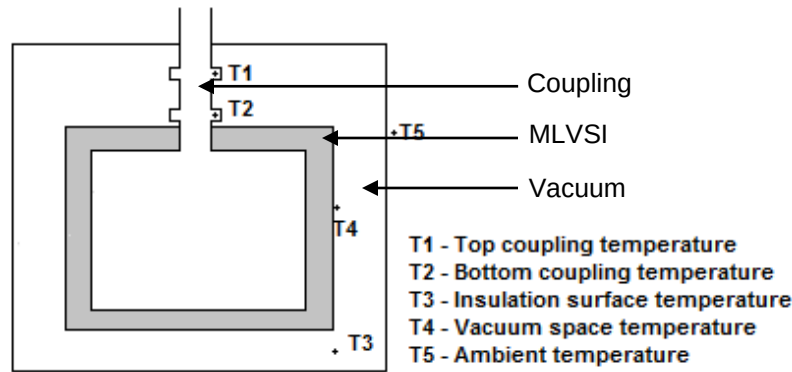


Figure 29: Schematic of temperature measurement positions

The top and bottom coupling temperatures were measured making use of two type T-thermocouples whereas three type K-thermocouples were used for the MLVSI surface temperature, the vacuum space temperature and the ambient temperature. These thermocouples were fitted through the wall of the outer vessel. Due to the small gaps between the thermocouple wire and insulation material, vacuum leaks were detected. This problem was resolved by using specially designed fittings involving thin copper wires which were fed through a stainless steel fitting filled with resin. Thermocouple wires were subsequently connected on both sides of the fitting to the copper wires. Thermocouples and sensors were calibrated before any measurements were taken. An analogue converter transferred the thermocouple resistance to a voltage signal which was sent to a computer and converted to temperature measurements. The analogue converter interface is shown in Figure 30.



Figure 30: Photo of the temperature analogue interface

The Scale Tronic Services, PRW model series is a high precision bench scale that was used for boil-off measurements. This scale has the unique capability to measure up to 30kg in 0.1g divisions ensuring the feasibility to measure gas boil-off accurately. The metal platform prevents the scale from being damaged by the cryostat (Fig. 31).

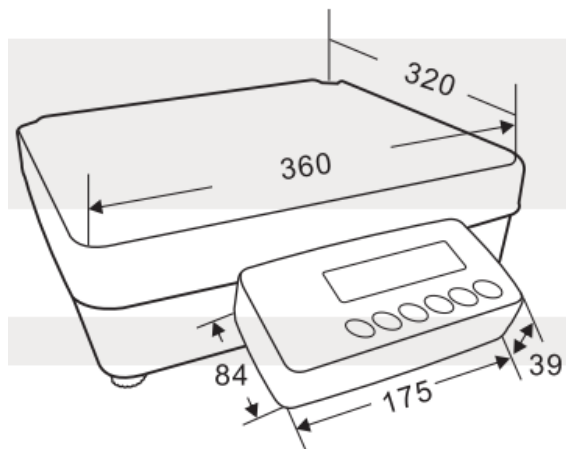


Figure 31: Schematic of the PWR high precision bench scale used to measure system weight loss (boil-off)

6.1 Configuration Testing Procedure

In order to test a MLVSI and/or coupling configurations, it was necessary to attach the inner tank to the stand and insulate it with a specific layer density of MLI. This insulation installation process, in order to eliminate dome edge effects, was discussed in Section 5.3. The insulated inner tank was then carefully removed from the stand and mounted to the bottom flange of the coupling using the copper seal. It was then mounted to the top supply flange of the outer tank dome. After connecting the thermocouples the inner tank was placed inside the outer tank and the assembly was closed up using the 8 hexagon bolts provided. The system was then ready for vacuum pumping and the vacuum sensor was used to indicate when the desired vacuum levels were reached. As soon as the system reached vacuum pressures below 2 mbar the gate valve was closed and the pipeline disconnected. By using the **fill pipe** and the scale reading, 3kg of liquid nitrogen was carefully poured into the cryogenic vessel.

In order to vent boil-off gas the small extraction fan connected to the pipeline was used (Fig. 25). The connected extraction fan was mounted on the now “empty” stand. For safety purposes another more powerful extraction fan was installed in the room and the electrical supply of these extraction fans was connected to the main power supply. Temperature and pressure measurements were taken every second out relayed through the analogue converter to the PC. When the vacuum level reached 6 mbar the vacuum pipeline was connected again between weight readings to generate the required vacuum level. The system’s weight was taken every minute and data saved throughout the testing period for later processing. The discussed procedure was followed for every test conducted as well as testing of the set-up prior to the scheduled experimental work.

6.2 Assembly Testing

In order to test the setup a configuration of ten layers of MLVSI was installed on the pressure vessel shaped inner tank and the performance based on nitrogen boil-off tested using the procedure described in section 6.1. The system was tested with a conventional straight inlet pipe configuration. This same configuration was tested the following day for a second time and a rapidly increased boil-off rate was noticed.

One could conclude that either the cryostat was not functioning properly or the insulation properties have changed. New insulation was installed and tested in the same manner to determine what caused the high boil-off rate. If the cryostat functioned properly, the test results should have been similar. Boil-off results obtained for both the old and the new installed insulation is shown in Figure 32.

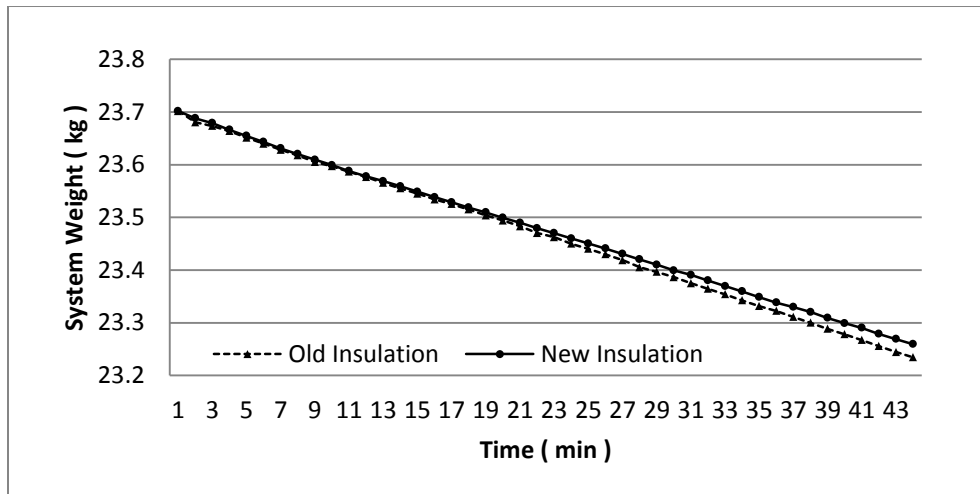


Figure 32: System weight vs. time indicating an increased boil-off weight for old insulation

The results obtained indicated a 6.5% variation in boil-off weight between the old and the new insulation for a period of 43 minutes and the following conclusion was made.

The system was designed to test MLVSI configuration samples for short periods; consequently the system only operated for a short period of time after which the gate valve was opened. The nitrogen still present in the system caused ice to form on the surface and between MLVSI layers due to the presence of moist air that entered the

system. As the system reached ambient temperatures after a few hours the ice melted forming moisture between alternative layers of the MLVSI which was absorbed by the glass fibre spacer material. This moisture acted as a direct conduction path between alternative layers of the MLVSI which decreased insulation performance when tested a second time.

To confirm that moisture did form between alternative layers the insulation was removed from the inner tank, weighed and kept in a dry place for several days. Subsequently a second weighing was carried out and a weight loss of 2.7 grams was established. The difference of 2.7 grams can only be ascribed to loss of moisture.

The deviation between alternative tests and the performance of the experimental system was further tested by conducting three tests with the same configuration as previously tested. All three tests were carried out for a period of 2 hours and the boil-off results obtained is shown in Figure 33. The system delivered very much the same boil-off measurements with every test conducted. A maximum variation of 0.5% was obtained over a testing period of 110 minutes which indicated that the experimental system was delivering reliable results. With the installation process known and the system operating as it should, the bellowed coupling configuration was now ready to be further evaluated.

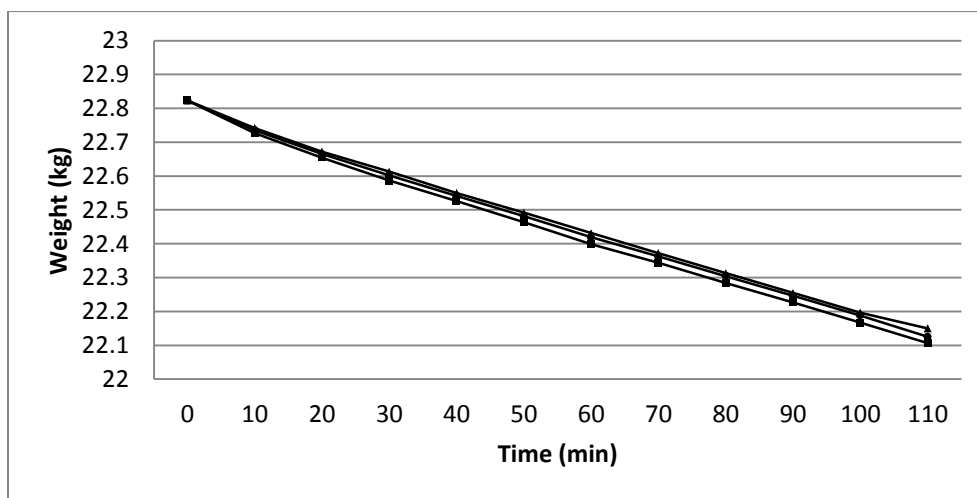


Figure 33: System weight vs. time illustrating deviations between alternative system setup tests

6.2.1 Inner Tank Coupling Testing

Before testing the bellowed coupling performance, another test was carried out without any coupling mounted between the inner and outer tank. The obtained results from this test were used to compare performances of later inlet coupling configurations. Figure 34 illustrates the reworked system test results to system weight vs. time variation for the conventional straight cylindrical inlet pipe.

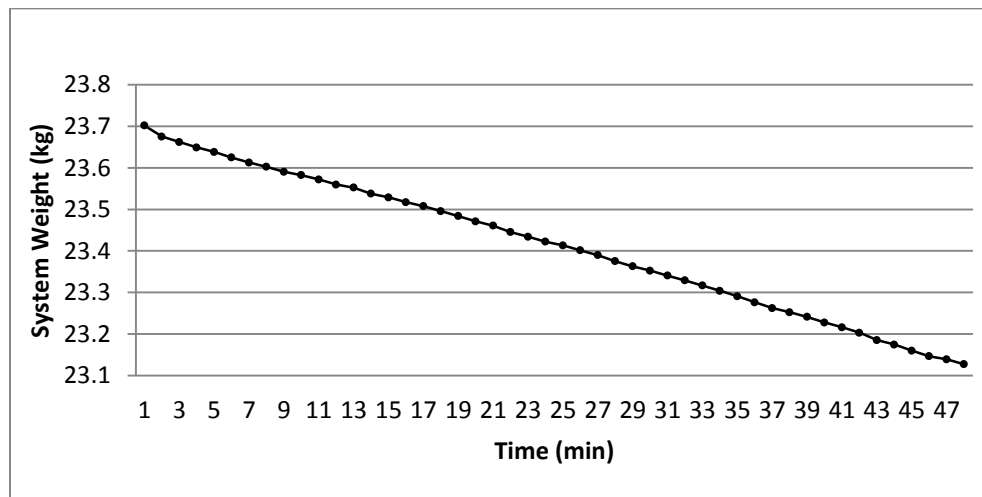


Figure 34: System weight vs. time for a conventional inlet pipe.

A system weight decrease from 23.7 kg to 23.1 kg was obtained for a period of 50 minutes, indicating that 600 grams of liquid nitrogen evaporated.

The designed bellowed coupling was mounted using the provided flanges welded to the inner and outer tank respectively (Fig 35). Thermocouples were fitted in 3mm Ø holes provided in the top and bottom flanges. The assembled system was again filled with liquid nitrogen as described and the boil-off as weight loss of the system determined during subsequent tests.



Figure 35: Photo of the coupling installed with top and bottom thermocouples shown

This coupling was tested and the following system weight vs. time measurements was obtained (Fig. 36).

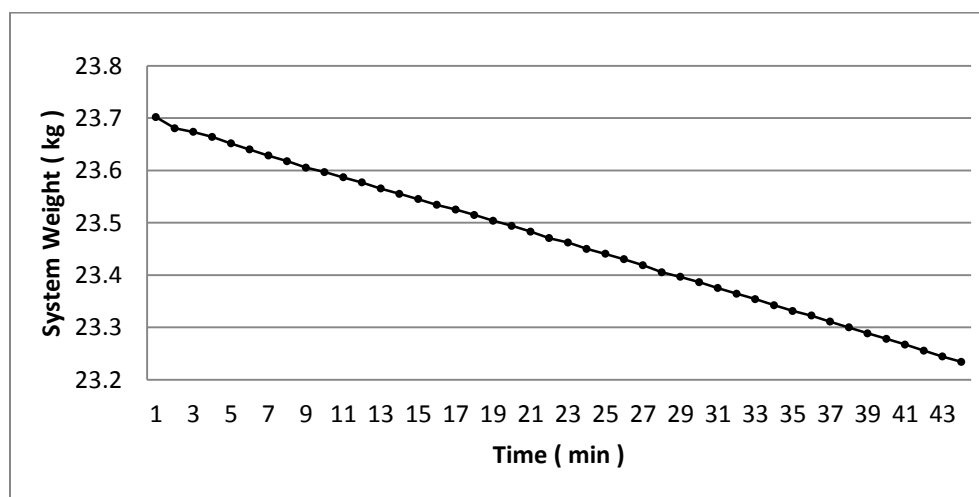


Figure 36: System weight vs. time measurement for the bellowed coupling

The system weight results obtained indicate that 21% (130 grams) less liquid nitrogen boiled off using the bellowed coupling. One could also make use of the boil-off rate processed from the system weight measurements to estimate if an improvement was made. This boil-off rate for both the inlet pipe as well as the bellowed coupling is illustrated in Figure 37.

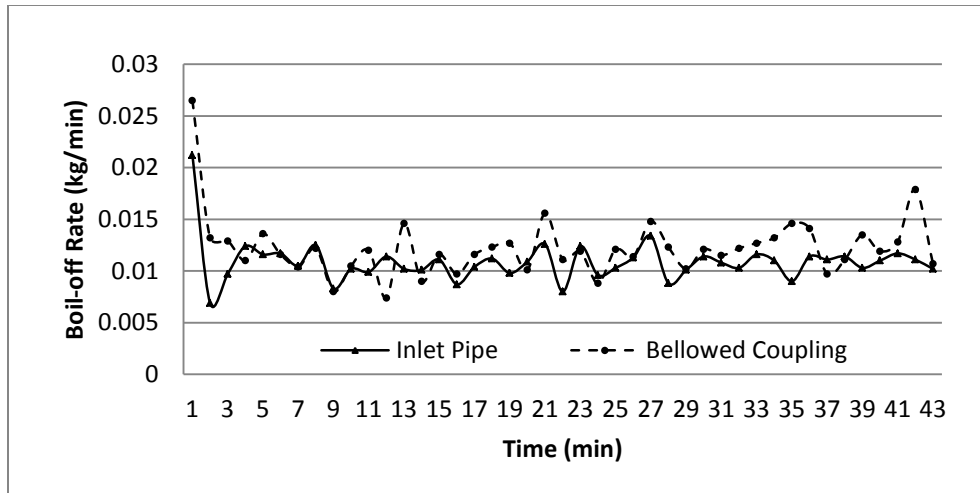


Figure 37: Liquid nitrogen boil-off rates for bellowed coupling and inlet pipe

The variance of boil-off rates is attributed to the constant vacuum pumping between experimental operations due to detected leaks. Future reference in this report refers to boil-off with respect to weight vs. time measurements of the filled system.

The measurements for T1 and T2 for both the inlet pipe and the bellowed coupling are presented in Appendix D. Temperature differences for these configurations were obtained and are presented in Figure 38

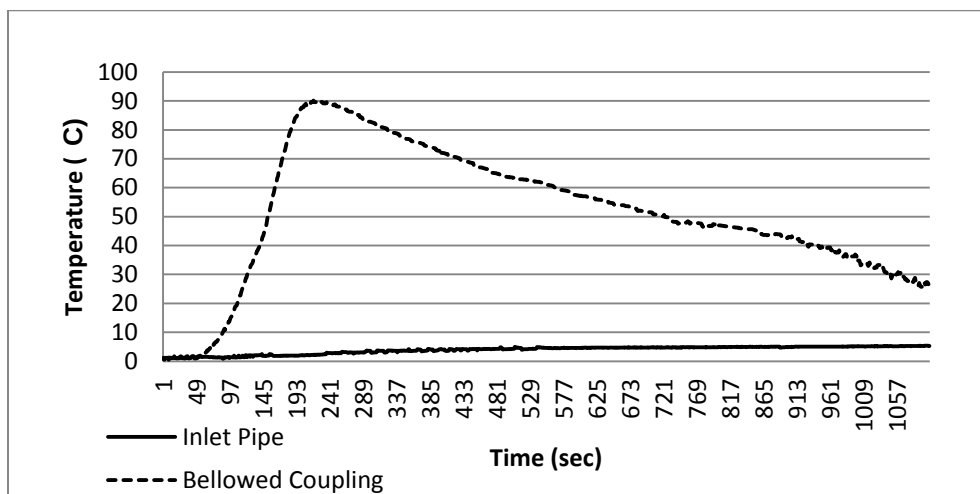


Figure 38: Graph of the temperature differences between the “hot” and “cold” sides of the inlet pipe and the bellowed coupling configuration

The temperature difference over the inlet pipe was almost constant throughout the testing period, indicating a higher heat transfer rate. The temperature difference for the bellowed coupling reached a maximum value in the first few minutes and then steadily declined as T1 dropped. From the results obtained it is concluded that the employed method to limit heat transfer by making use of a thin walled bellow delivered better performance although boil-off rates were still high.

The MLVSI surface temperature (T4) was plotted vs. time and shown in the Figure 39. The insulation surface temperature decreased from about -32°C in the first 250 seconds. From this point further on the temperature alternated between temperature differences of 10°C .

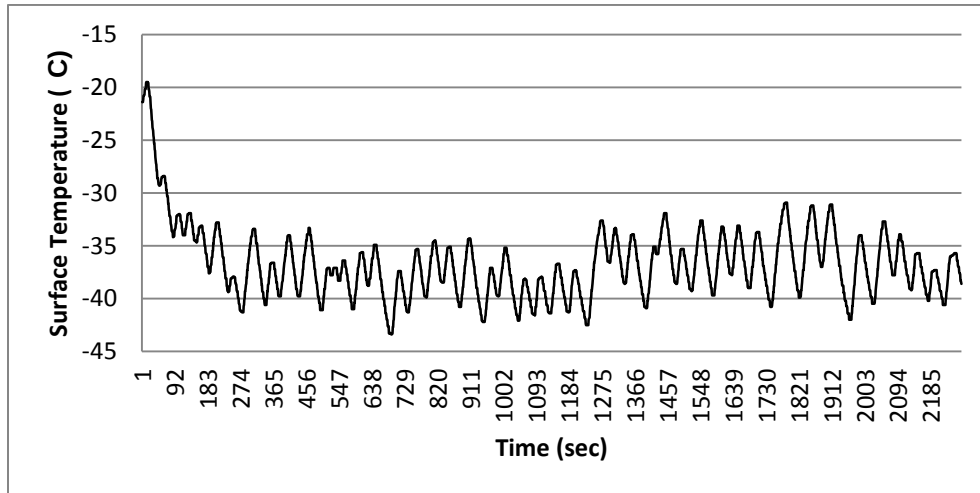


Figure 39: MLVSI surface temperature

The specific shape of the curve can be attributed to the vacuum pump constantly being switched on and off to keep the desired vacuum level. This also indicates the effect vacuum levels had on the surface temperature of MLVSI. It was concluded that for every 1 mbar vacuum lost, the insulation surface temperature would rise with 2.5°C to 3°C . This means that at a vacuum pressure of 6 mbar the tank's insulation surface temperature was 10°C more than at 2 mbar which may drastically influence heat transfer to the cryogenic fluid.

Chapter 7: Assembly Modifications

7 Background

This chapter reports on modifications applied to the inlet/outlet coupling design in effort to further reduce boil-off. To further investigate heat transfer through the coupling an ANSYS finite element analysis (FEA) was carried out. The results of this analysis are reviewed and the resulting additional modifications to the coupling are presented. Modifications with respect to the MLVSI installation procedure involving tank geometry changes to further reduce edge effects and boil-off are presented.

7.1 Coupling Modifications

7.1.1 Coupling Modification A

Analysis of the bellowed coupling indicated that heat transfer through the bellowed coupling could be reduced by reducing the cross sectional area and the heat flux path of the bellow. Since space requirements excluded further lengthening of the bellow it was decided to reduce the cross sectional area thickness of the bellow from 0.4 mm to 0.25 mm. A CAD model of the modified coupling (Coupling A) is shown in Figure 40.

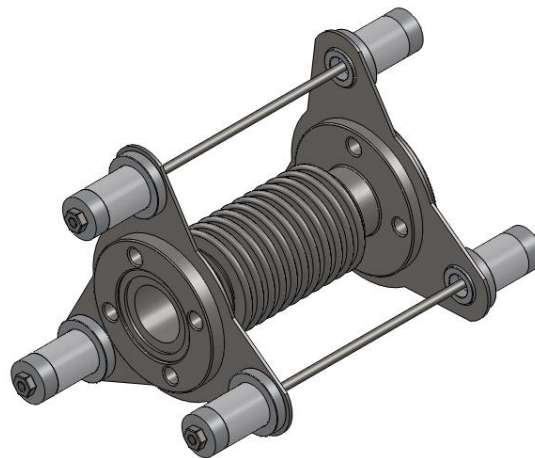


Figure 40: A CAD model of the assembled Coupling A

Due to the reduction in material thickness of the bellow (from 0.4mm to 0.25 mm) the circumferential stresses would have been too high when carrying the inner tank load.

This problem was addressed by implementing a support system to handle the weight of the inner tank without adding unnecessary heat flow paths to the system (Fig. 40). An exploded view of the modified design is shown in the Figure 41.

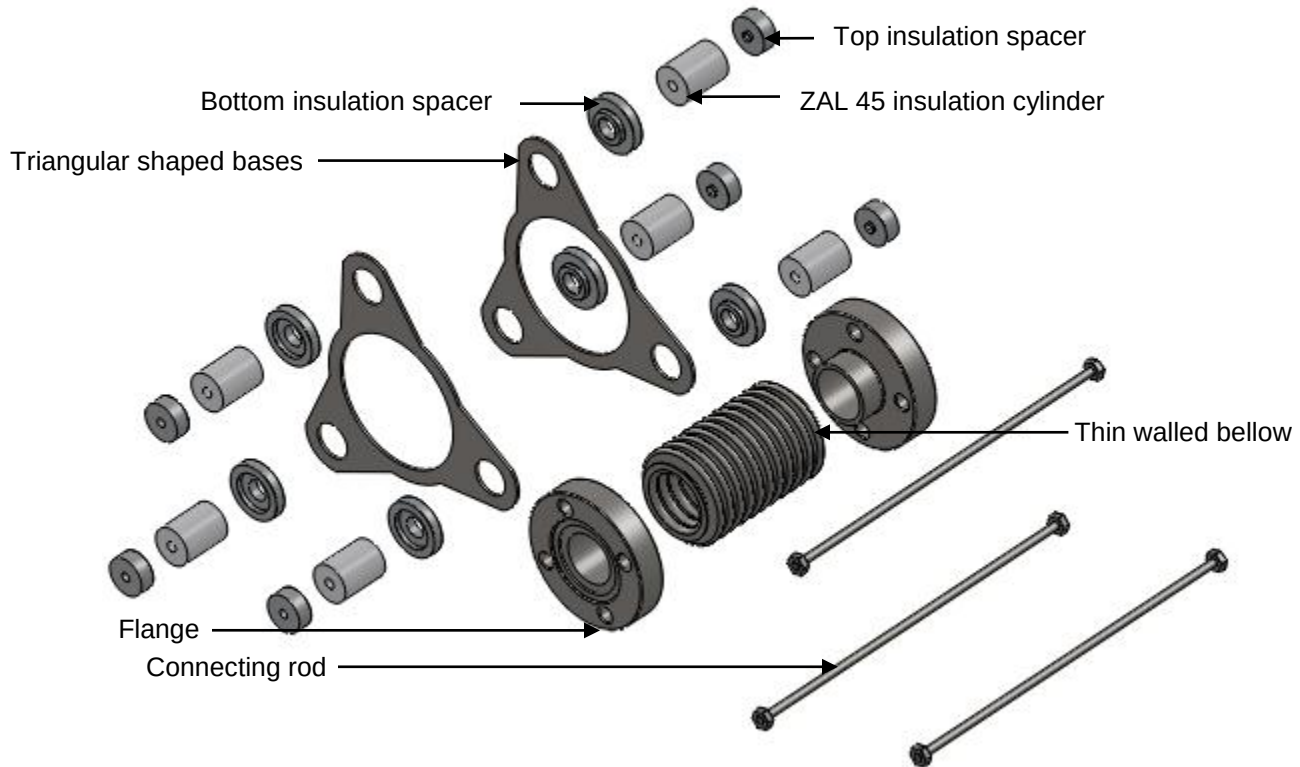


Figure 41: Exploded view of the CAD model of Coupling A

Coupling A was produced from a SAE 316L (0.25 mm) stainless steel bellow and the support system consists of 3 mm diameter stainless steel rods and two triangular shaped bases. The triangular bases were manufactured from 2 mm thick stainless steel plates using a CNC milling machine and were welded to the flanges using tungsten inert gas welding. The use of the rods would have caused unacceptable heat transfer between flanges when welded directly to the triangular bases giving rise to the use of ZAL 45 fibrous alumina insulation (Thermal Ceramics Inc., with a thermal conductivity of 0.23 W/m.K at 100 °C) to manufacture small cylindrical insulation spacers to limit this heat transfer. Due to the fragile nature of this insulation material aluminium spacers were required to align these cylindrical insulation spacers and the support rods (Drawing CRYO - IN 0011, Appendix F).

The two column supports were aligned perfectly to ensure an equal stress distribution through the ZAL 45 insulation and the support rods on loading. The envisaged heat transfer paths through the modified supports are illustrated in the cross sectional view shown in Figure 42.

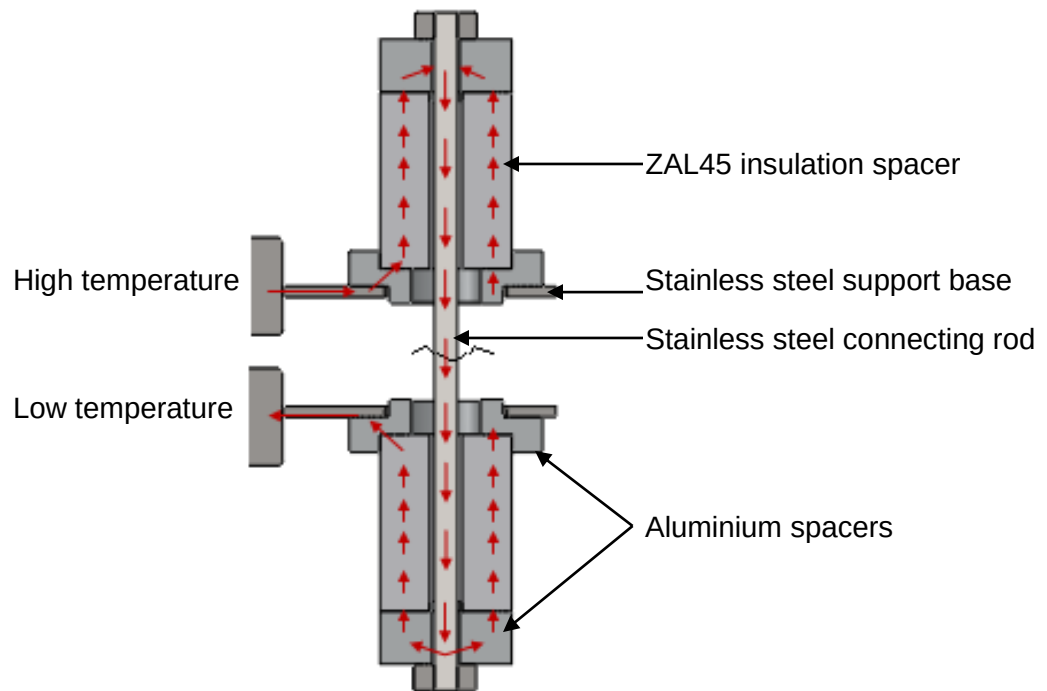


Figure 42: Heat flow path through modified column support

The ZAL 45 insulation as well as the bottom spacer has larger inner diameters than the connecting rods to avoid direct contact that would otherwise defeat the purpose. The top and bottom spacers position the connecting rod so that the rod does not touch the inner sidewall of the insulation nor the bottom spacer material. Heat is transferred through the connecting rod to the top spacer then back through the ZAL 45 insulation to the bottom spacer from where it is then transferred through the support base to the bottom flange. An illustration of the modified assembled coupling (Coupling A) is shown in Figure 43.

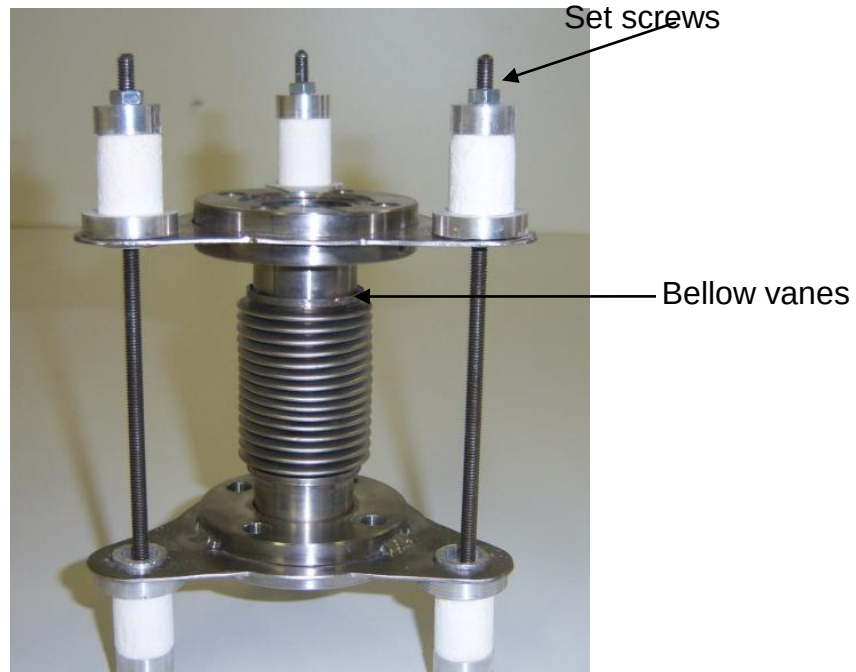


Figure 43: Photo of the manufactured and assembled coupling A

The alignment as well as the axial stability of Coupling A was set using the connecting rod set crews. The connecting rods were set to a specific length to ensure that the bellows vanes did not touch which otherwise could lead to heat being transferred as a result of conduction between alternative bellows vanes.

Coupling A was tested in the manner described in Section 6.2 and results obtained (Data presented in Appendix D) indicate that a 32% (190 grams) reduction in boil-off was realised when compared to the conventional straight inlet nozzle systems test data.

7.1.2 Coupling Modification B

Coupling A was further modified by installing high conductivity copper plates to the bellow to act as heat sinks. These copper plates were manufactured in a concave dome form and were installed between the vanes of the thin walled bellow (Fig 44).

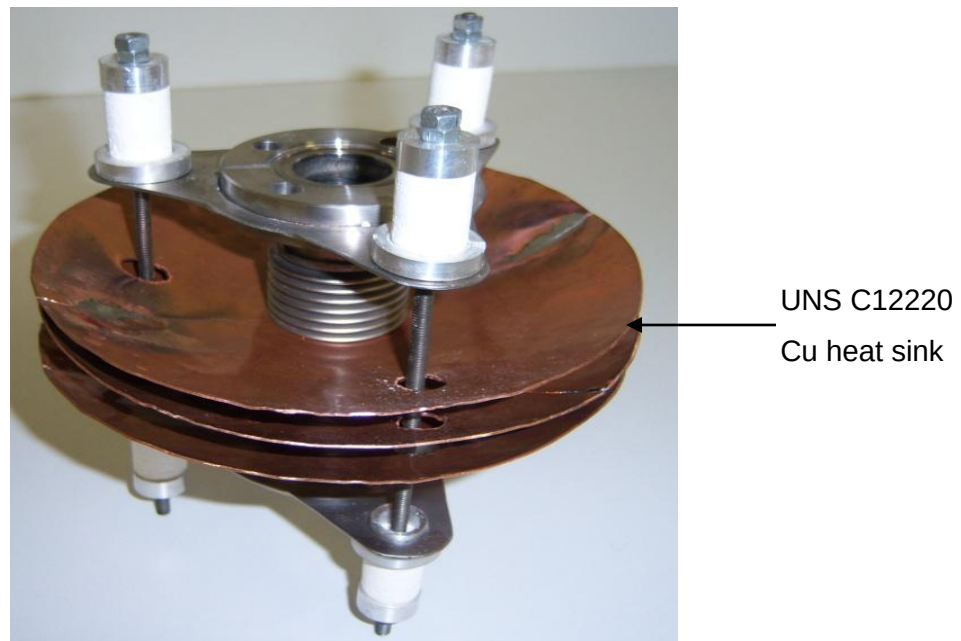


Figure 44: Photo of the manufactured and assembled coupling B

This assembly of heat sinks is composed of two halves per disc which were soldered to each other and to the bellow. The copper heat sinks were provided with 3 x 10mm Ø holes spaced triangularly to allow the protrusion of the coupling connecting rods and to eliminate heat transfer through contact with the rods. The domed discs had a bright surface finish to reduce radiation effects.

This coupling (Coupling B) was tested in the manner described in Section 6.2. The results of this configuration test indicate that a 38 % (230 grams) reduction in boil-off weight was obtained when compared to the results of the conventional inlet pipe systems test (Data presented in Appendix D).

7.1.3 Coupling Modification C

To determine if heat transfer due to radiation between the bellow and the surrounding external tank could be reduced, a modification was made to Coupling B. A schematic of this modification to Coupling B is presented in Figure 45.

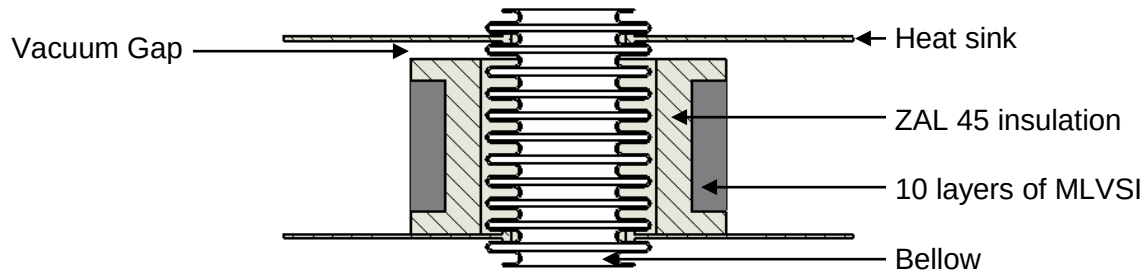


Figure 45: A schematic illustration of a ZAL 45 part for coupling C to limit radiation

For this purpose a cylindrical insert (85mm diameter with a circular recess to a depth of 12 mm) was machined from ZAL 45 insulation board to accommodate 10 wrapped layers of MLVSI. To accommodate this modification (Coupling C) one of the copper heat sink discs was removed (Fig.46)

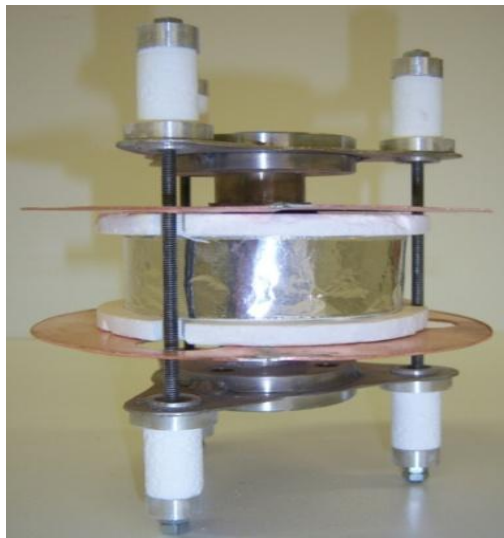


Figure 46: Photo of the manufactured and assembled Coupling C with wrapped MLVSI to limit radiation to the bellow.

Coupling C was tested in the same manner as before. The data obtained indicate that by using the Coupling C configuration a 45% (270 grams) reduction in boil-off weight, when compared to using a conventional inlet pipe, can be realised. However, it was found that the use of the ZAL 45 base material in this configuration (Coupling C) was prone to fracturing when handled on completion of test runs. This phenomenon can possibly be ascribed to the freeze fracturing during service due to residual moist contained in interconnected pores typical of this type of fibrous insulation material.

7.1.4 FEM Coupling Steady State Simulation

In order to further optimise the design of Coupling B, the heat transfer through the assembly was modelled with the aid of FEM using the ANSYS code (ANSYS). In addition, a FEM was also applied to the assembly to find the optimum positions of the copper heat sinks.

The material properties presented in Table 4 were assigned to the components of the Coupling B CAD model imported to ANSYS as required for simulation purposes.

Table 4: Thermal properties for components used in the FEM model

Component	Isotropic Conductivity ($\text{Wm}^{-1}\text{C}^{-1}$)	Specific Heat ($\text{Jkg}^{-1}\text{C}^{-1}$)
Stainless steel	15.1	480
ZAL 45 insulation	1.4	750
Aluminium	114	875
Copper	400	385

This coupling configuration was simulated using a boundary temperature difference of 40°C between the top and bottom flanges of the coupling.

Figure 47 illustrates a colour distribution of the modelled heat flux through Coupling B. These results indicate a maximum heat flux through the support plates and the aluminium spacers. In other words more heat was transferred through the support columns than the bellow itself and therefore confirms the design consideration and the effective reduction in heat surface area that would inhibit conductivity. These results indicate that heat flux through the coupling can be further reduced through selection of support rod material with a lower conductivity.

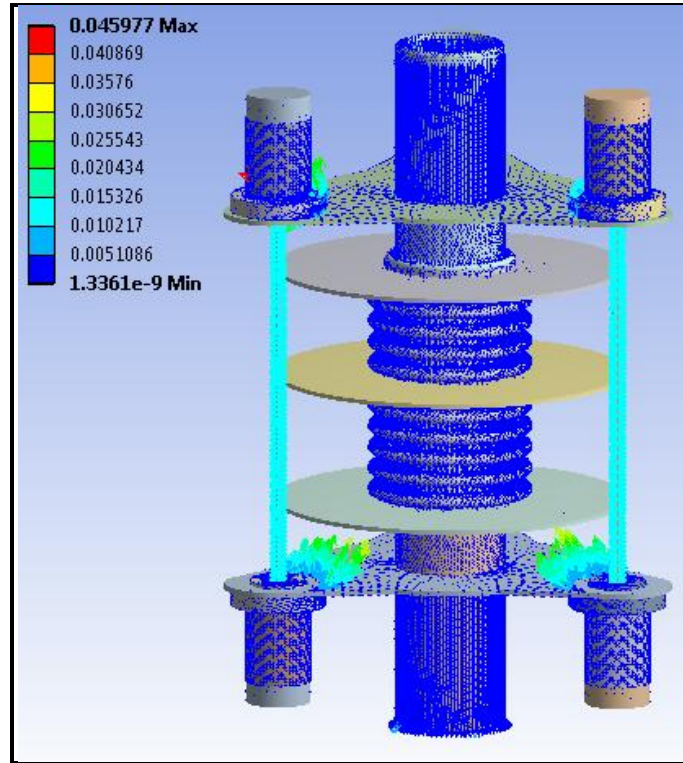


Figure 47: FEA result of the heat flux through Coupling B

By taking note of the temperature colour legend (Fig. 48) of both Figure 48a and Figure 48b it is clear that the temperature difference Δt , between the top and bottom insulation spacers of Coupling C (glass fibre composite) has increased compared to that of the Coupling B configuration (with stainless steel rods). Furthermore, software probes applied to both ANSYS simulations (Fig 48a and 48b) show that the support rods had a temperature of -53°C underneath the middle copper plate for Coupling C whereas for Coupling B this temperature was determined to be -46°C indicating an “insulation” improvement of 13°C .

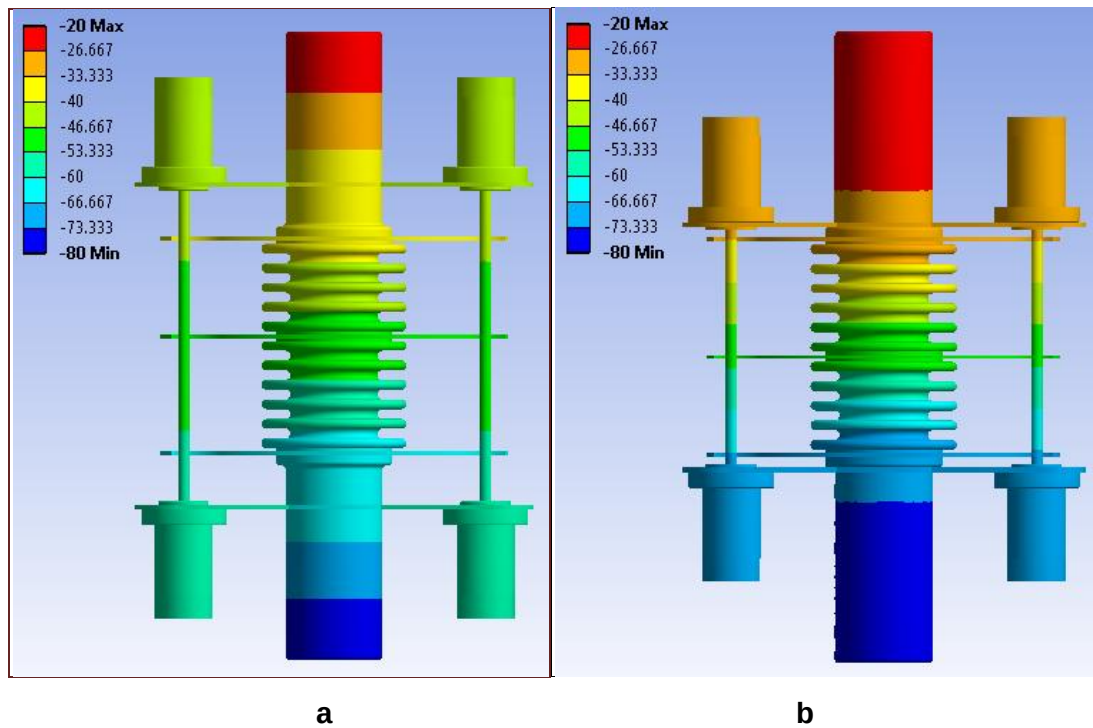


Figure 48: FEA of the steady state temperature distribution (°C) through modified couplings: a) stainless steel columns and b) glass fibre columns

Additional simulations were run to determine the optimum positioning of the installed heat sinks. Three simulations were run on modified models (based on the Coupling B configuration) with the copper heat sinks placed at various vertical positions on the bellow. Results obtained for each of these simulations are presented in Figure 49.

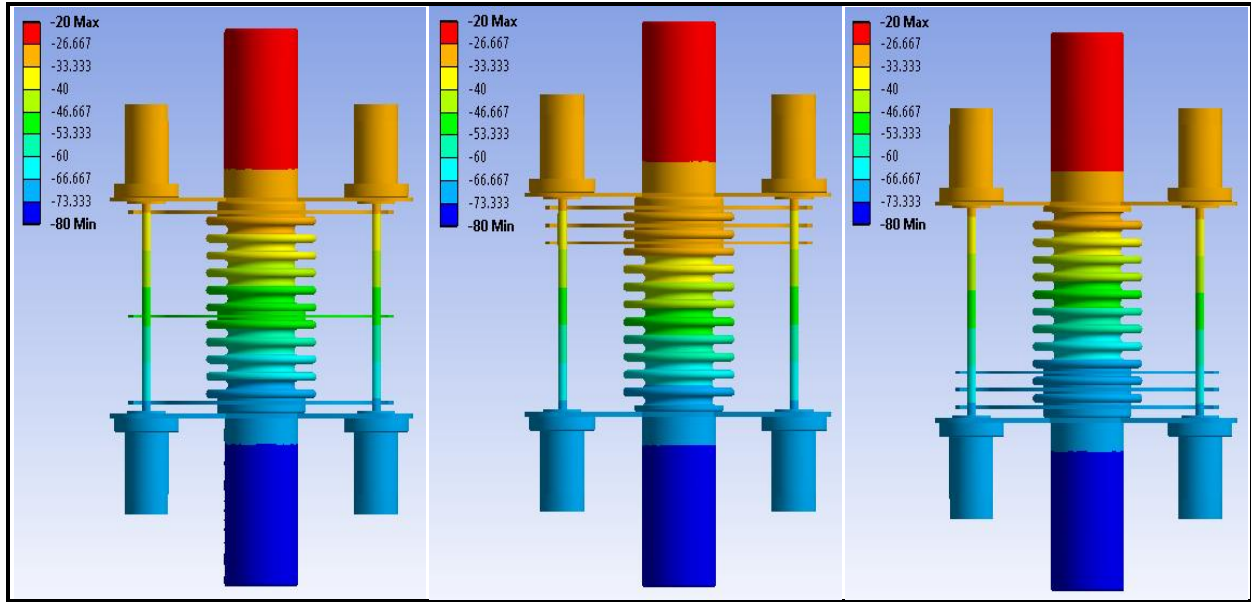


Figure 49: Temperature distribution through Coupling B for various copper heat sinks positions; a) Evenly spaced, b) evenly spaced on the high temperature side, c) evenly spaced on the low

The results of software controlled temperature probes placed in the centre of the bellow circumference of each temperature distribution simulation result (Fig. 49 a, b and c) are shown in Table 5.

Table 5: ANSYS probe temperatures for heat sink configurations of Coupling B

Position of heat sink discs	Evenly Spaced	High Temperature Side	Low Temperature Side
Maximum Temperature (°C)	-20	-20	-20
Minimum Temperature (°C)	-80	-80	-80
Probe Temperature (°C)	-51	-45	-54

The model of Coupling B simulated in this manner, returned a circumference probe temperature of -51°C when the copper heat sinks were evenly spaced. A temperature of -54°C was returned with the copper heat sinks placed at the low temperature side of this

coupling configuration. It is thus concluded that for optimum system performance the heat sinks should be evenly spaced on the low temperature side of the bellow.

7.2 MLVSI Application Modifications

The effective geometry of the inner tank was changed with the aim to limit installation time. It was also aimed at reducing heat transfer through the spherical shaped headers.

7.2.1 Elimination of Edge Effects by Changing Tank Geometry

In this section a modification to further reduce heat transfer edge effects resulting from the required application technique of MLVSI over the pressure vessel header sections, is presented.

The inner tank outside geometry was changed by using ZAL 45 insulation end caps to flatten the dome surfaces in order to get an external cylindrically shaped pressure vessel (Fig 50) that would simplify the application of the MLVSI over the headers.

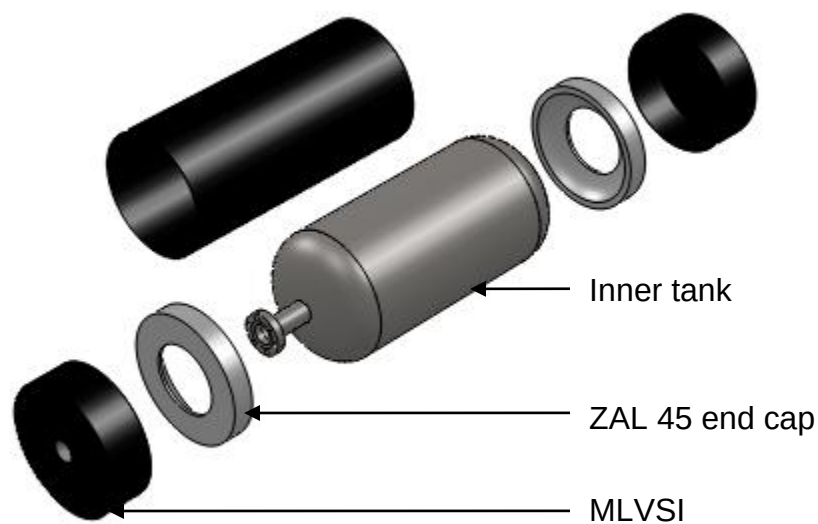


Figure 50: Exploded view of a CAD model of the inner tank and the ZAL45 insulation end caps

An illustration of the assembly procedure is shown in Figure 51a, b and c.

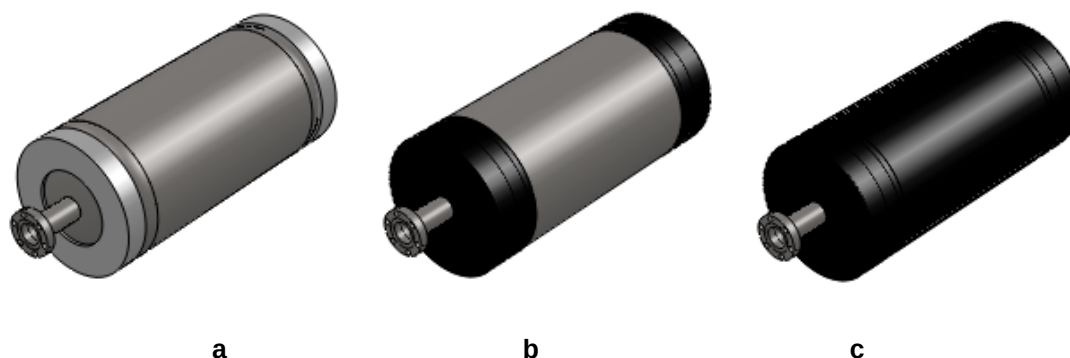


Figure 51: CAD model illustration of end cap and MLSVI installation to a pressure vessel

ZAL 45 end caps with the same diameter as the inner tank were machined and were placed at the ends of the inner tank (Fig.51a). MLI was then cut in circular discs that overlapped the diameter of vessel. These discs were then laid over the end caps and then cut along radials to the circumference of the vessel in order to fold the cut segments as required underneath the MLI as rectangular shaped MLVSI layers were applied(Fig.51a,b).

The boil-off test results (Fig. 52) obtained show that this modification had no effect on boil-off rate over a period of 45 minutes.

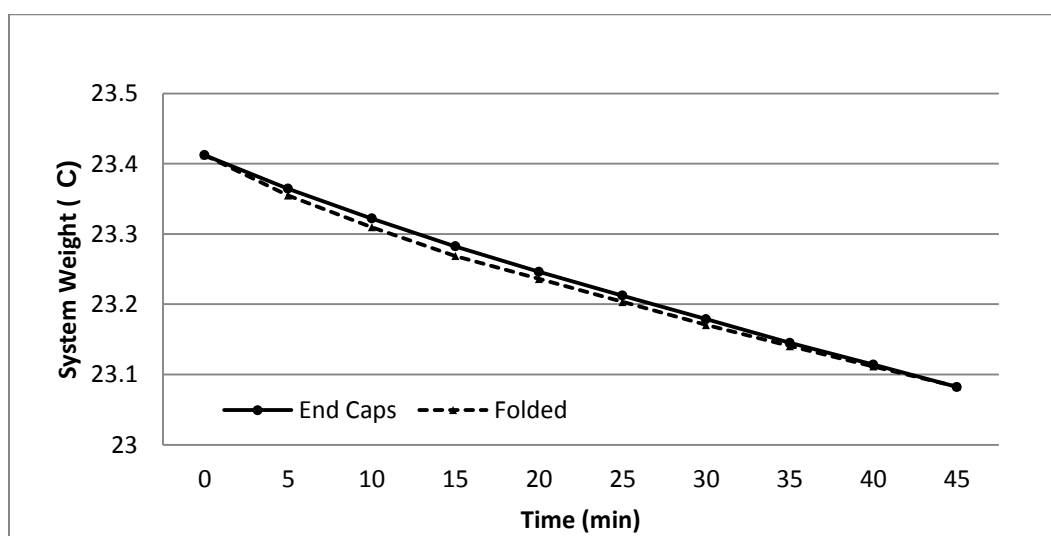


Figure 52: System weight vs. time for folding and end cap configurations respectively

7.3 Discussion

A graph showing the comparative system weight (boil-off) of the cryogenic systems with different inlet/outlet coupling configurations (Straight inlet pipe, Coupling A and B) is presented in Figure 53. All tests were conducted with a MLVSI layer density of 10.

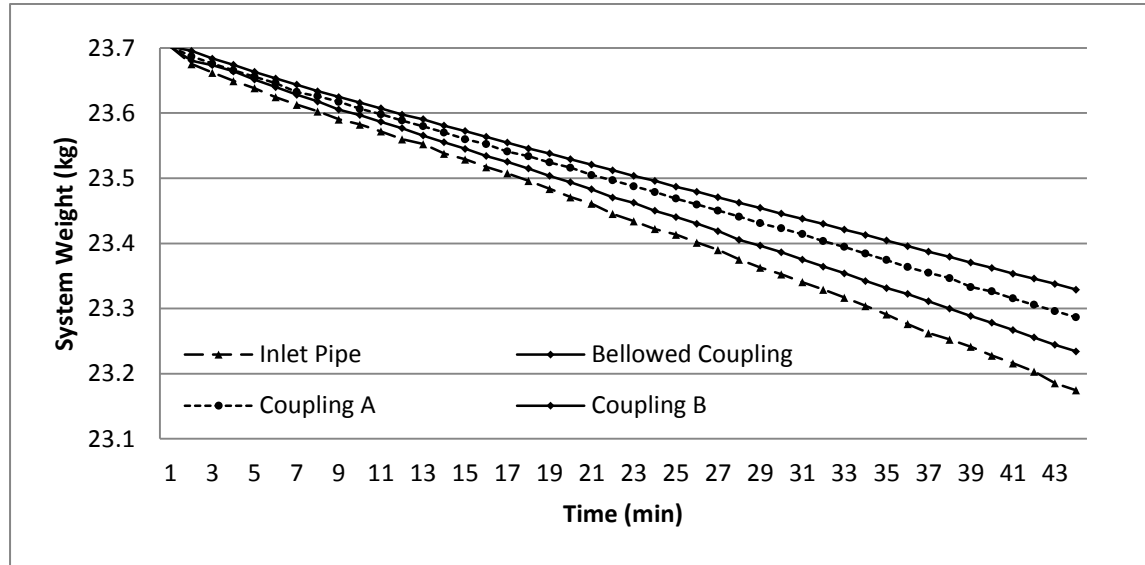


Figure 53: System weight vs. time for different coupling configurations tested

Table 6 presents the reduction in boil-off weight for the different coupling configurations calculated from the data of Figure 53.

Table 6: Boil-off improvements for modified couplings

No	Connection	Boil-off Weight (g/45min)	Weight saved (g)	Reduction with respect to base (1) (%)
1	Inlet Pipe	600	-	-
2	Bellowed Coupling	470	130	23
3	Coupling A	410	190	32
4	Coupling B	370	230	38

A total improvement of 38% (230 g) was obtained by making use of Coupling B rather than a conventional inlet pipe. Thus 5.5 kg liquid nitrogen would be saved per day by using Coupling B with concave copper heat sinks installed to the thin walled bellow. ANSYS simulations carried out show that by replacing the support rods with a glass fibre composite with a thermal conductivity and a specific heat of $1.4 \text{ Wm}^{-1}\text{C}^{-1}$ and $750 \text{ Jkg}^{-1}\text{C}^{-1}$ respectively an additional 25 per cent with respect to circumferential bellow temperature can be saved. ANSYS simulations furthermore show that the heat sinks need to be evenly spaced at the bottom of the bellow as was implemented in the Coupling B configuration.

By changing the tank geometry to a cylindrical shape the same system weight loss as the method used in Section 5.2 was obtained. This modified method of application of MLSVI proved to be less time consuming and that it may be employed on a spherical pressure vessel without jeopardizing or increasing boil-off.

Chapter 8: Optimum System

8 Introduction

In this section all modifications that had a positive impact to reduce boil-off and that were finally integrated in one single system and tested to evaluate the performances of selected redesigned coupling and MLVSI configurations, are presented.

8.1 Optimum System Performance

A model of the optimum coupling is shown in Figure 54.

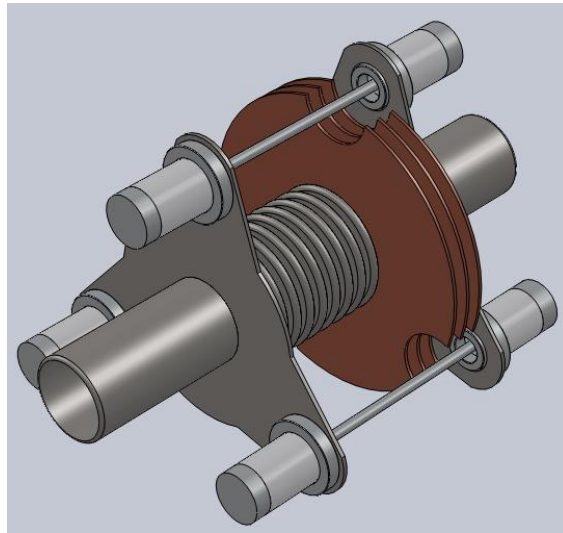


Figure 54: Model of optimum coupling

The shape of the copper heat sinks were however modified to the shape shown in Figure 54 but were installed as concluded in the ANSYS simulation carried out to find the optimum positioning on the bellow at the low temperature side of the coupling - that is nearest to the cryogen vessel. For this test the stainless steel supports rods were replaced by 3mm diameter fiber glass. The coupling (Coupling D) was assembled and installed as shown in Figure 55.



Figure 55: Photo of the manufactured and assembled optimised coupling (Coupling D)

A layer density of 25 was applied to the inner tank using the method discussed in Section 5.2.

A test was conducted for a boil-off period of two hours to enable the system to reach steady state conditions. The temperature difference between the top and bottom sides of Coupling D measured is plotted in Figure 56.

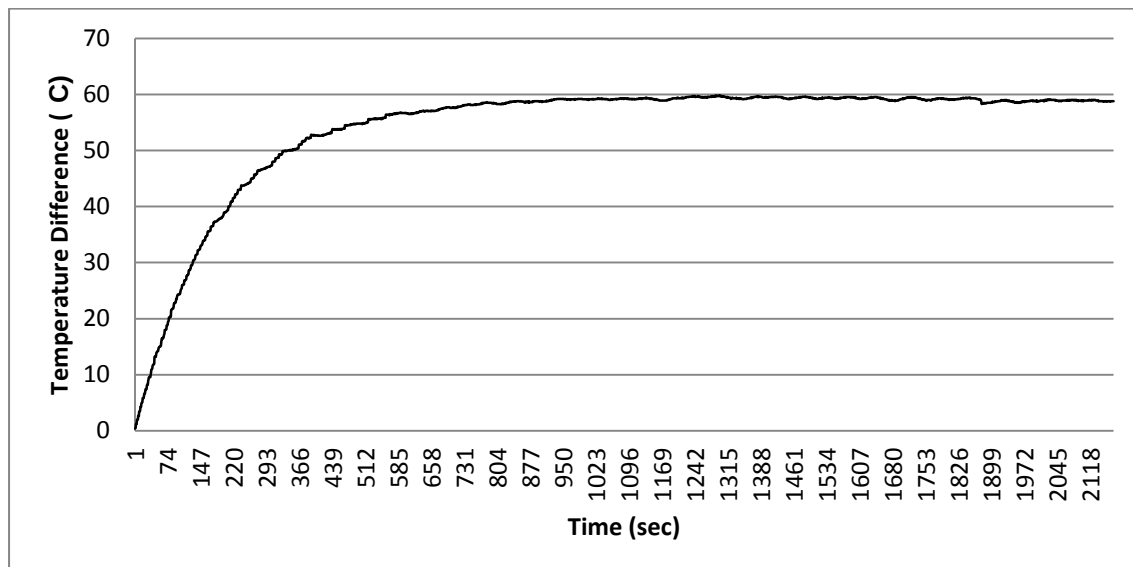


Figure 56: Temperature difference over the optimised coupling (Coupling D)

A temperature difference of 60°C was obtained over Coupling D which indicated a 25% increase in Δt between the lower and upper parts of this coupling compared to the Δt generated when using the Coupling B configuration during configurations tests.

The system weight measurements vs. time representing boil-off for the optimized system are shown in Figure 57.

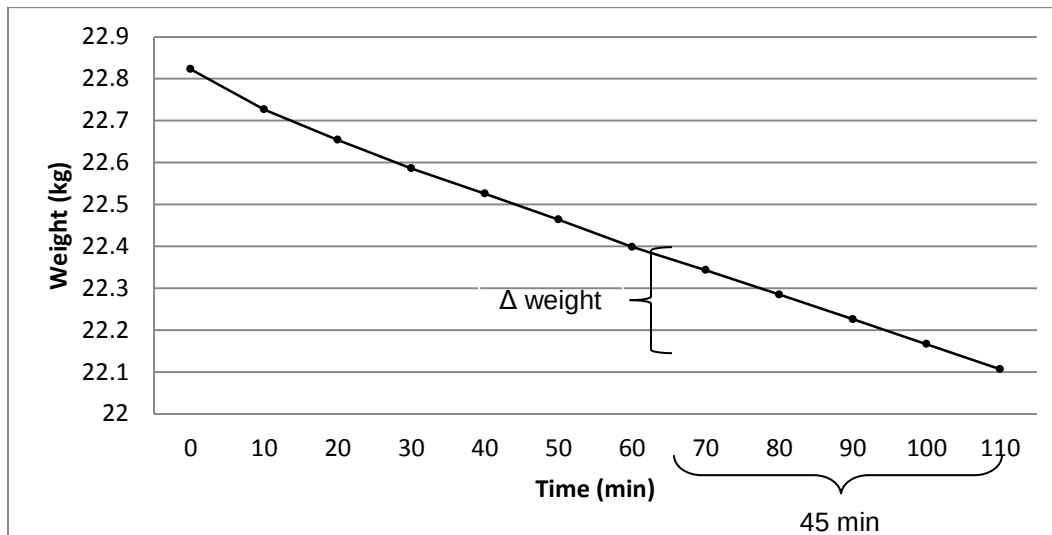


Figure 57: Optimised System Weight vs. Time Measurement

The results indicate that a 250 grams boil-off for a period of 45 minutes has taken place (Figure 57). This was a 58% (350grams) decrease in system weight indicating that 8.5 liters of liquid nitrogen were saved per day with respect to the conventional inlet pipe. One should keep in mind that this test was conducted with 25 layers of MLVSI instead of the 10 used for other tests and can also be attributed to the lower boil-off weight.

Figure 58 gives the boil-off rate (kg/10min) vs. time and illustrates that it takes 80 minutes for the system to reach steady state conditions. The amount of liquid nitrogen evaporated per day can also be calculated using this boil-off rate graph. After steady state conditions were reached the boil-off rate was approximately 0.06kg/10min. This indicates that when the system reaches steady state conditions the nitrogen loss will be 360g per hour (what the boil-off will be with liquid hydrogen is still not known)

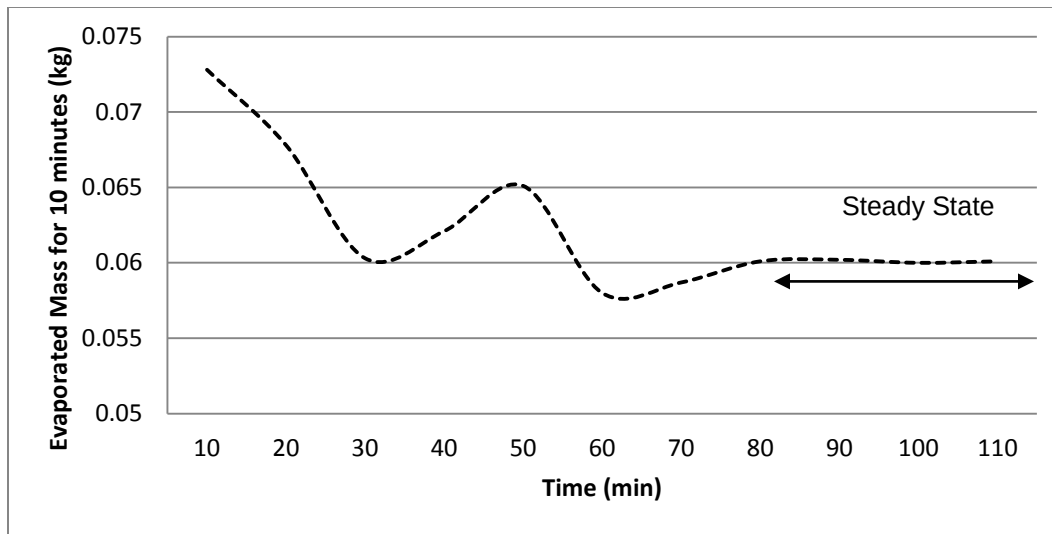


Figure 58: Evaporated mass (boil-off) for ten minute increments vs. time showing how steady state conditions is reached

Chapter 9: Conclusions

The application of MLVSI to cryogenic storage systems is the most promising insulation for these applications. MLVSI performance goes hand in hand with coupling performance and together these two critical components can limit boil-off of gas.

A method used to insulate the spherical header, with MLVSI, of a typical pressure vessel by making use of a jig to cut the insulation before application, was tested and documented. This method delivered promising results although it was a very time consuming installation process. Another method was implemented where the spherical headers of such a pressure vessel was covered with ZAL 45 material in order to achieve a cylindrical surface without changing the actual pressure vessel geometry. This method was tested and the same boil-off weight was obtained as for the previous method although the installation process was less time consuming.

The conventional inlet pipe used in cryogenic systems to limit heat transfer between the inner and outer tanks was replaced with a specially designed bellowed coupling and tests indicate that a 21% reduction in boil-off weight with respect to the inlet pipe was obtained. This bellowed coupling was improved by further decreasing the bellow wall thickness and a 32% improvement was obtained with respect to the conventional inlet pipe.

Further modifications were made by installing concave copper discs between alternative bellow vanes in order to act as heat sinks and limit heat transfer. This coupling showed a remarkable boil-off weight decrease of 38% with respect to the inlet pipe.

The above mentioned coupling was simulated by making use of a FEM using ANSYS code and it was found that it would be possible to further improve the coupling by replacing the support bases and the rods with a glass fibre composite material. It was also found that for best coupling performance the copper heat sinks had to be installed at the low temperature side of the bellow.

An optimum system was assembled based on the findings of the test results and this system was tested. By using a thin walled bellow with glass fibre supports and copper heat sinks placed at the low temperature side of the coupling, a 58% boil-off weight decrease was obtained with 25 layers of MLVSI, compared to the conventional inlet pipe with 10 layers of MLVSI. The change in layer densities had a minor influence on boil-off rates.

This lead to a total weight of 8.5 kg liquid nitrogen being saved with respect to the conventional inlet pipe tested.

During the conduction of tests it was found that constant vacuum pressures play an important role in MLVSI performance. If vacuum pressures alternates during testing of this layered material the surface temperature of the MLVSI is highly effected which leads to high heat transfer rates and boil-of rates.

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Appendix A: Theoretical Model

Theoretical Model Used for Testing Various Insulation Materials.

The spherical thin walled container used to calculate boil-off rates for liquid nitrogen using different insulation material is shown in Figure 68. The container has a diameter of 0.5 m and an insulation thickness of 25 mm. The nitrogen is kept at 77K and the container has a vent for boil-off purposes and the ambient air is at 300K. The heat transfer rate and the nitrogen boil-off are calculated for every type of insulation. Results are shown in Table 7.

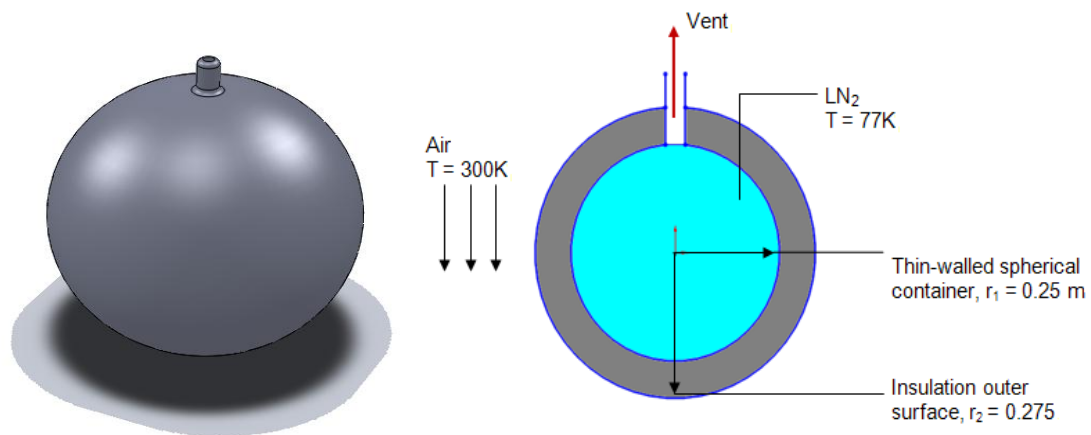


Figure 59: Thin-walled insulated nitrogen tank

Table 7: Boil-off rate for various Insulation materials used to insulate a thin walled nitrogen container

Insulation Material	Thermal Conductivity (W/mK)	Heat Transfer (W)	Boil-off rate (l/day)	Boil-off time day(s)
Perlite powder	0.4	1785	958.9	0.07
Polyurethane Foam	0.17	1001	537.7	0.12
Hollow Glass Spheres	0.047	333.7	179.3	0.37
Expanded Perlite	0.036	260.4	139.9	0.47
Air	0.024	177.2	95.22	0.69
Extruded Polystyrene (R-12)	0.023	170.1	91.41	0.72
Glass Microsphere (100 torr)	0.018	134.3	72.17	0.91
Aerogel (10 torr)	0.008	60.77	32.65	2.01
Silica Powder in a Vacuum	0.0017	13.06	7.018	9.33

Cylindrical MLVSI winding machines

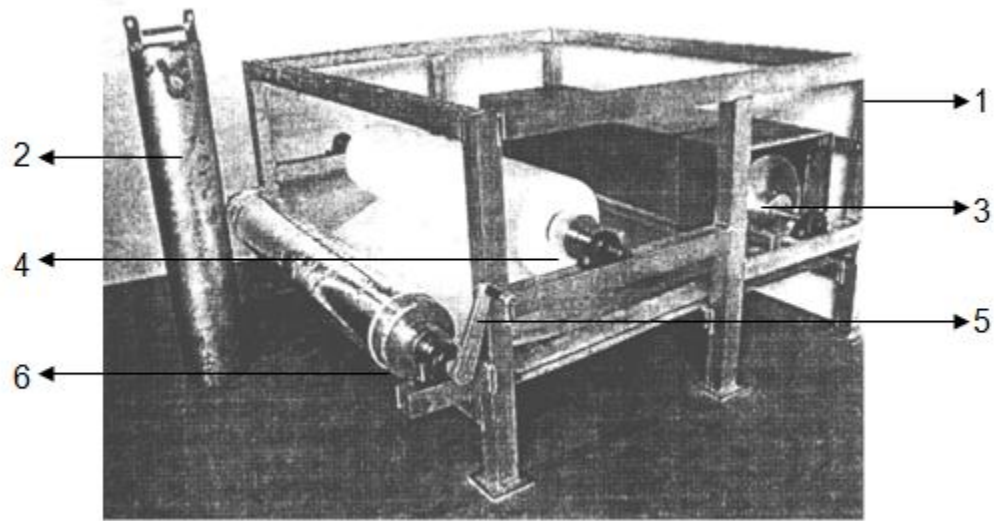


Figure 60: Multi-Layer Insulation Wrapping Machine (NASA) [25]

- | | |
|------------------------|--------------------|
| 1) Frame | 4) Spacer material |
| 2) Sleeve | 5) Handle |
| 3) Metalized film roll | 6) Copper sleeve |

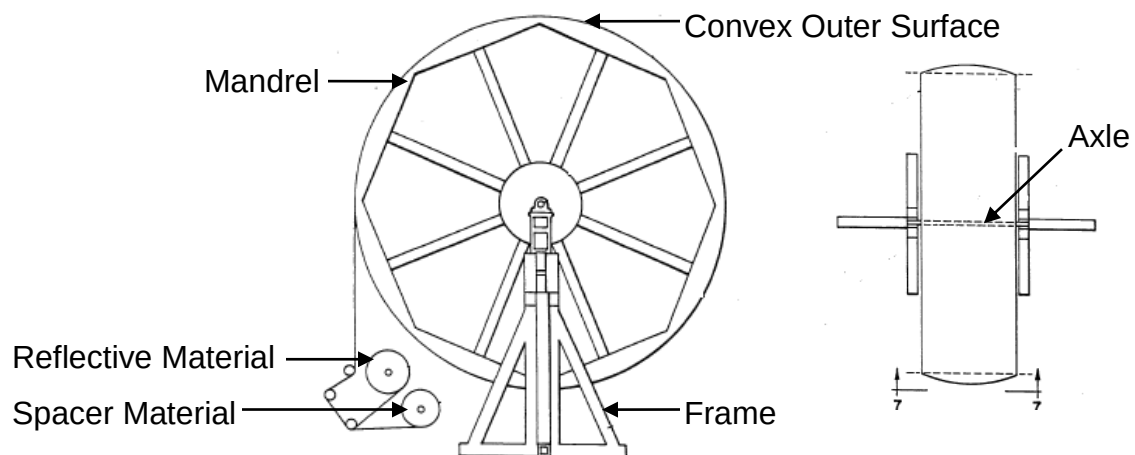


Figure 61: Fabricating Apparatus for Multilayer Insulation Blankets [29]

Appendix B: Safety

Liquid Nitrogen and Its Health Effects

On April 1883 nitrogen was first liquefied at the Jagiellonian University by Zygmunt Wróblewski and Karol Olszewski. It is produced by fractional distillation of liquid air. Liquid nitrogen boils at 77 K (–196 °C) at atmospheric pressure which causes rapid freezing when in contact with living tissue. It is odourless, tasteless, colourless and even not irritating. This means it has no warning properties which make it very dangerous to work with. When nitrogen is boiling off in a closed space it will replace the air to levels lower than required by a human being. It has been discovered by OSHA that 19.5% oxygen is the minimum concentration of air to survive. When inhaling large amounts of nitrogen with limited oxygen, symptoms like dizziness, vomiting, loss of consciousness and death may occur. Death is usually a result of bad judgment which leads to unconsciousness that prevents self rescue. According to the University College Dublin safety office the oxygen concentration following a spill can be determined using the equation [19]:

$$\frac{100 \times V_0}{V_r}$$

Where $V_0 = 0.209(V_r - V_g)$

V_r is the volume of the room in m³

V_g is the volume of the liquid in m³ x expansion coefficient – 682

Cryostat Safety Requirements

Disobeying safety rules can result in injuries such as cold burns and frostbite when body parts get in contact with cryogenics. Some other injury incidents that can occur is when gas vaporises and there is a sudden volume change which can cause the vessel to burst violently [20]. The reason for this is because of the instant volume expansion with a factor up to 1000. Consequently, cryogenic liquids require specialized storage containers. In addition to the above safety risks mentioned, Stewart et.al is of the opinion that structural embrittlement is also a hazard at these low temperatures. Recommended safety wear when handling cryogenic equipment is as follows:

- Cryo-gloves
- Face Shield
- Safety Goggles
- Lab Coat
- Long Pants

This PPE (personal protection equipment) is recommended by the Department of environmental Health and Safety. Furthermore, all metal jewellery has to be removed from hands and wrists when filling such cryostat or dewar. The cryogen must not splash or left unattended and in the event of a cold burn the following should be kept in mind [19].

Any restrictive clothing should be removed, but not from the frozen tissue. Flush the affected area with tap water less than 40 °C to return to normal body temperature. Heat should not be applied to affected area and do not rub and cover with a loose, sterile dressing until medical assistance.

Appendix C: Calculations

Calculations for percentage boil-off per day for various insulation materials. The program EES (Engineering Equation Solver) was used for calculations.

"Spherical, thin walled metallic container"

```

T_in = 77
r_in = 0.25
t = l/1000
T_out = 300
h_air = 20
r_out = r_in + t
rho_N = 804
h_fg = 2 * 10^ 5
{k = 0.0017}
l = 25

Rt_cond = 1/(4*pi*k) * (1/r_in - 1/r_out)
Rt_conv = 1/((h_air * 4 * pi)*r_out^2)
R_tot = Rt_cond + Rt_conv
deltaT = (T_out - T_in)
q_1 = deltaT/R_tot

q_1 = m_dot_ps * h_fg
m_dot_pd = m_dot_ps * 3600 * 24
V_1 = (m_dot_pd / rho_N) * 1000
P_days = (V_volume/V_1)
V_volume = ((4/3)*pi*r_in^3)*1000

Z = (V_1/V_volume) * 100

```

" Liquid nitrogen"
 "Radius of inside container"
 "Silica powder insulation thickness"
 "Ambient air"
 "Convective coefficient of air"
 "Radius of outside container"
 "Nitrogen density"
 "latent heat of evaporation"
 "Evacuated silica conduction"
 "l/day"
 "days to total boil-off"
 "percentage per day of total capacity"

Results

	1	2	3	4
	k [W/mK]	q ₁ [W/m ²]	V ₁ [%]	P _{days} [days]
Run 1	18	4113	2210	0.02962
Run 2	0.4	1785	958.9	0.06825
Run 3	0.17	1001	537.7	0.1217
Run 4	0.047	333.7	179.3	0.365
Run 5	0.036	260.4	139.9	0.4678
Run 6	0.024	177.2	95.22	0.6873
Run 7	0.023	170.1	91.41	0.716
Run 8	0.018	134.3	72.17	0.9069
Run 9	0.008	60.77	32.65	2.005
Run 10	0.0017	13.06	7.018	9.327

The following *boil-off vs. insulation* graph shows how gas boil-off decreases as insulation thickness increases.

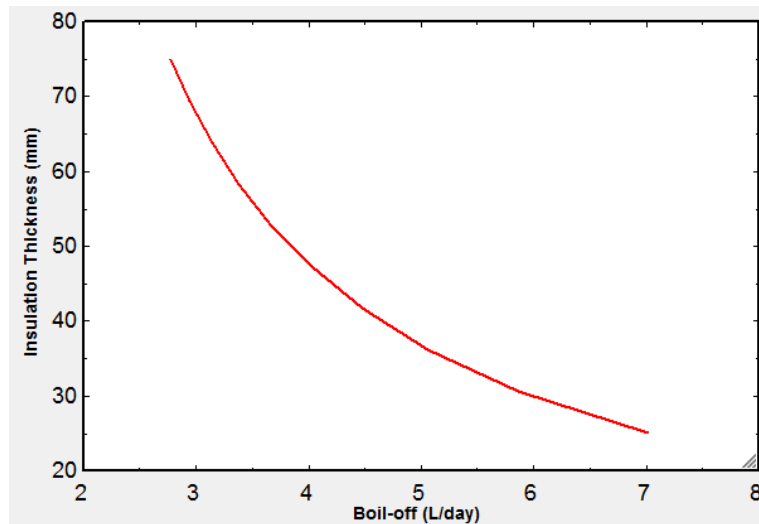


Figure 62: Gas Boil-off vs. Insulation Thickness

The following *boil-off vs. tank radius* graph shows how gas boil-off decreases as the tank radius increases.

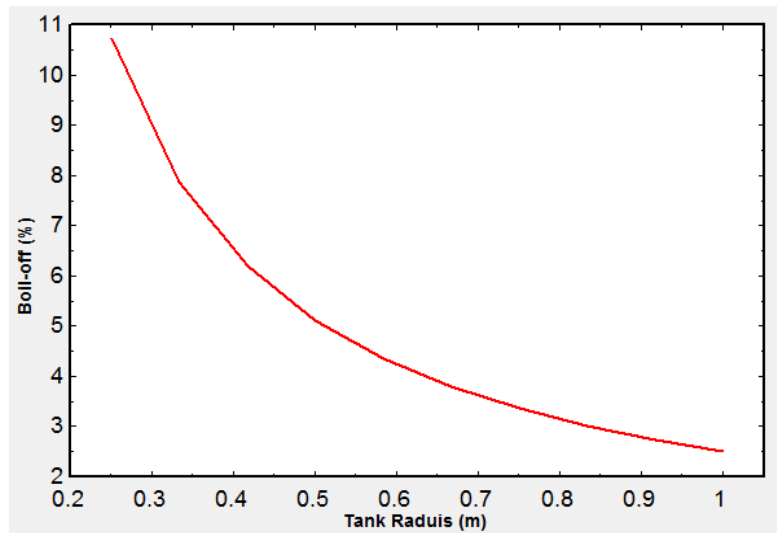


Figure 63: Gas Boil-off vs. Tank Radius

Calculations for determining the cryostat's inner and outer tank dimensions.

"Inner Tank 1....."

"Variables"

$$\rho = 808.607$$

$$r_1 = 0.08$$

"Spherical Volume"

$$V_{sp,1} = ((4/3)) * \pi * (r_1^3)$$

"Cylinder Volume"

$$V_{sil,1} = \pi * (r_1^2) * h_{sil,1}$$

$$V_{tot,1} = V_{sp,1} + V_{sil,1}$$

$$V_{tot,1} = 0.005$$

"Weight Nitrogen"

$$M_1 = \rho * (V_{tot,1} - (V_{sp,1}/2))$$

"Outer Tank 2....."

"Variables"

$$t = 0.0223$$

$$s = 0.020$$

$$r_2 = t + s + r_1$$

$$h_{sil,2} = h_{sil,1} + a$$

$$a = 0.060$$

"Insulation Thickness"

"Vacuum Space"

"Aerogel Thickness"

"Spherical Volume"

$$V_{sp,2} = ((4/3)) * \pi * (r_2^3)$$

"Cylinder Volume"

$$V_{sil,2} = \pi * (r_2^2) * h_{sil,2}$$

$$V_{tot,2} = V_{sp,2} + V_{sil,2}$$

Results

$$a = 0.06 \text{ [m]}$$

$$\rho = 808.6 \text{ [kg/m}^3\text{]}$$

$$t = 0.0223 \text{ [m]}$$

$$V_{sp,2} = 0.007662 \text{ [m}^3\text{]}$$

$$h_{sil,1} = 0.142 \text{ [kJ/kg]}$$

$$r_1 = 0.08 \text{ [m]}$$

$$V_{sil,1} = 0.002855 \text{ [m}^3\text{]}$$

$$V_{tot,1} = 0.005 \text{ [m}^3\text{]}$$

$$h_{sil,2} = 0.202 \text{ [kJ/kg]}$$

$$r_2 = 0.1223 \text{ [m]}$$

$$V_{sil,2} = 0.009493 \text{ [m}^3\text{]}$$

$$V_{tot,2} = 0.01715 \text{ [m}^3\text{]}$$

$$M_1 = 3.176$$

$$s = 0.02 \text{ [m]}$$

$$V_{sp,1} = 0.002145 \text{ [m}^3\text{]}$$

Appendix D: Experimental Data

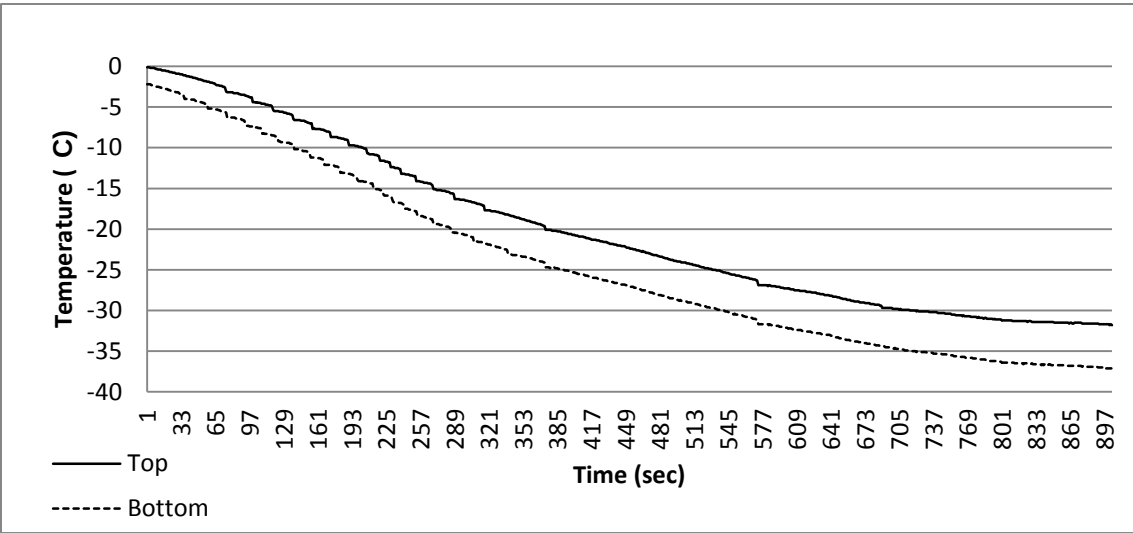


Figure 64: Top and bottom coupling temperatures vs. time for inlet pipe

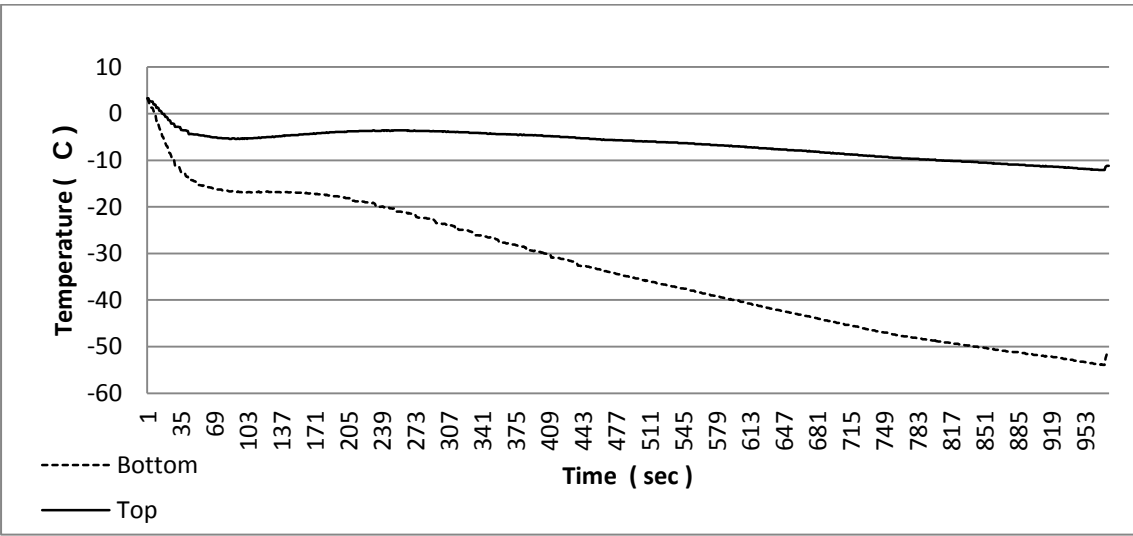


Figure 65: Top and bottom coupling temperatures vs. time for coupling

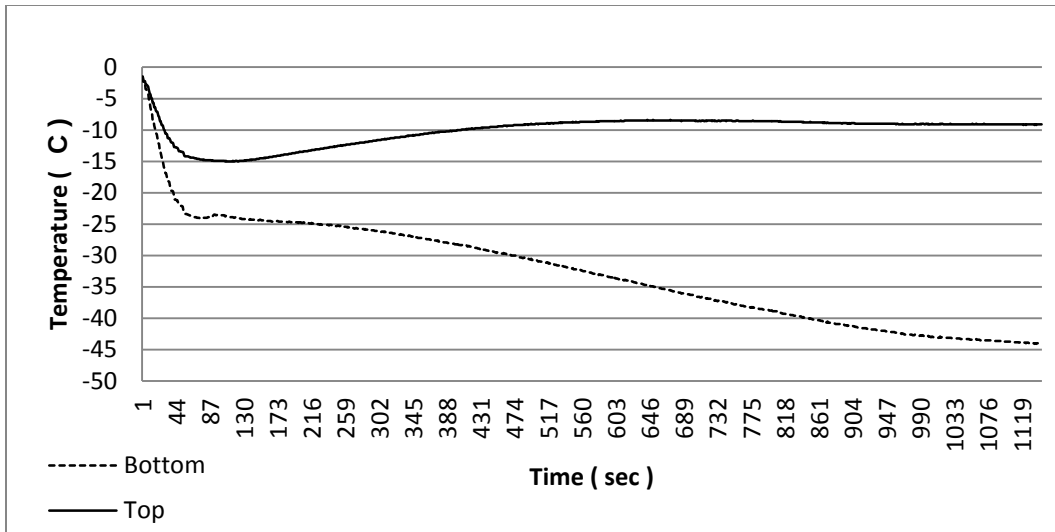


Figure 66: Top and bottom coupling temperatures vs. time for improved Coupling A

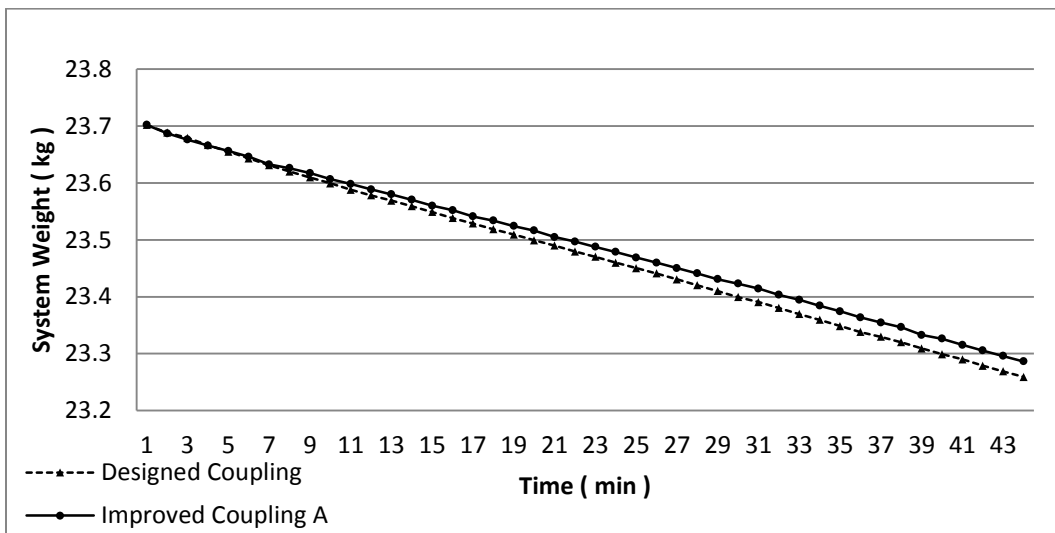


Figure 67: System weight vs. time for the coupling compared to the improved Coupling A

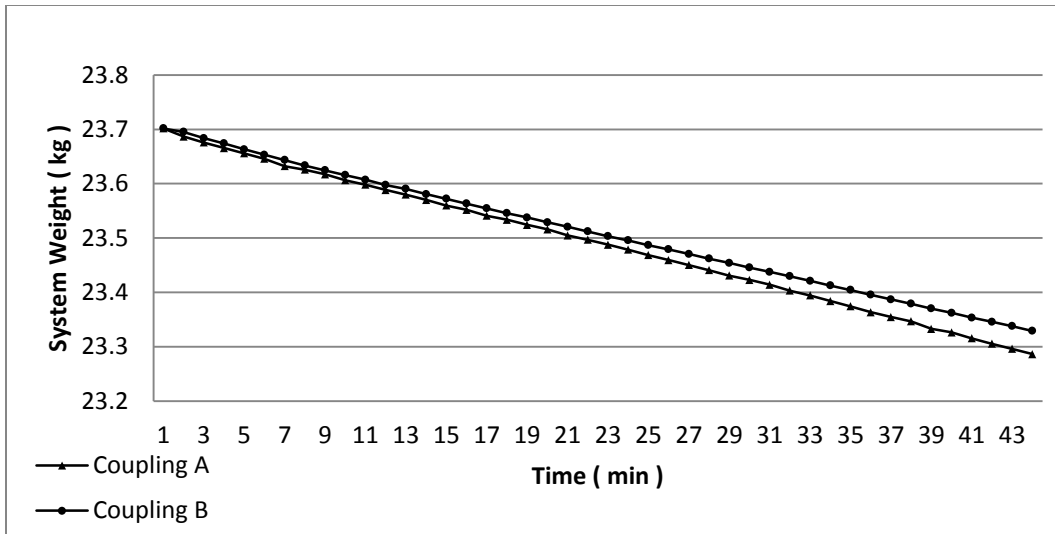


Figure 68: System weight vs. time for coupling A compared to Coupling B

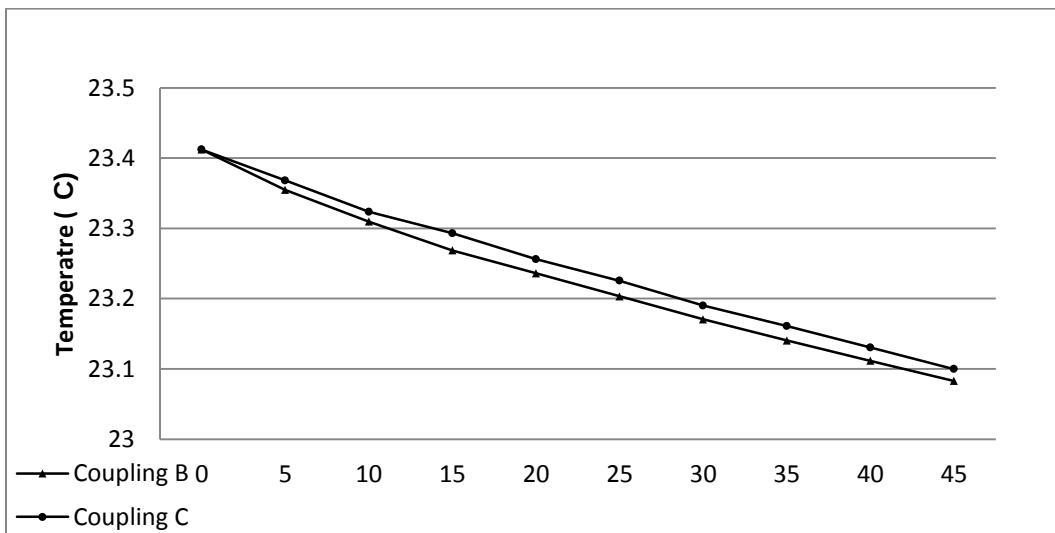


Figure 69: System weight vs. time for coupling B compared to Coupling C

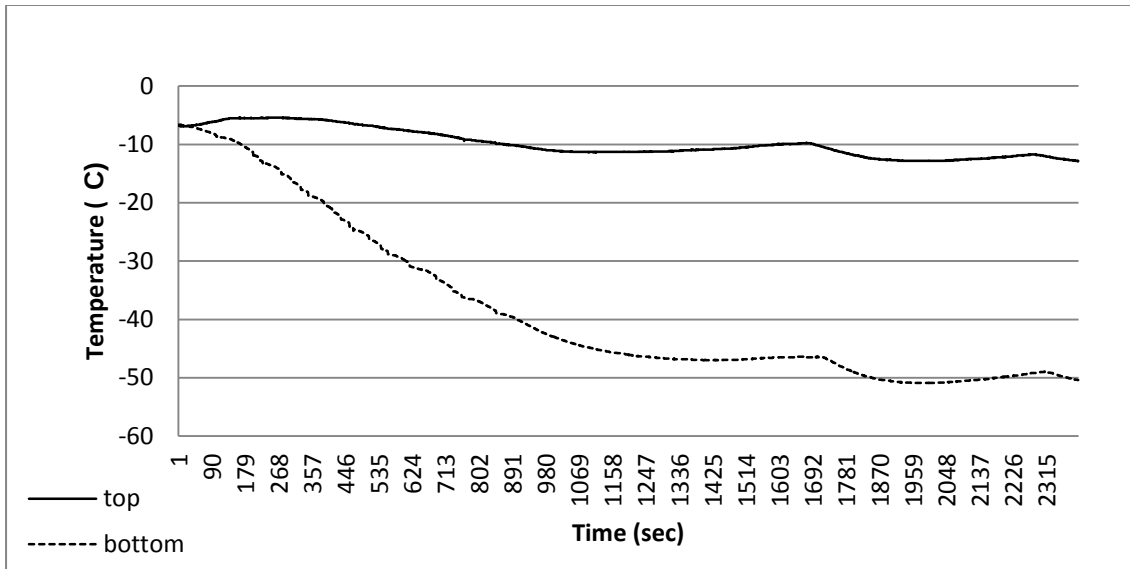


Figure 70: Top and bottom coupling temperatures vs. time for ZAL 45 insulation

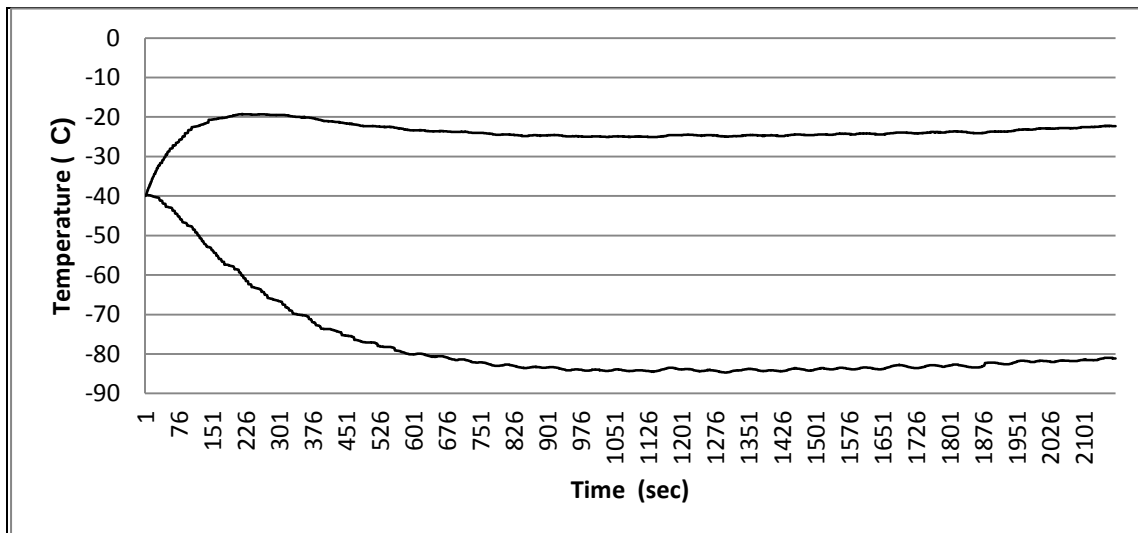


Figure 71: Top and bottom coupling temperatures vs. time for optimum system

Appendix E: Part List and Suppliers

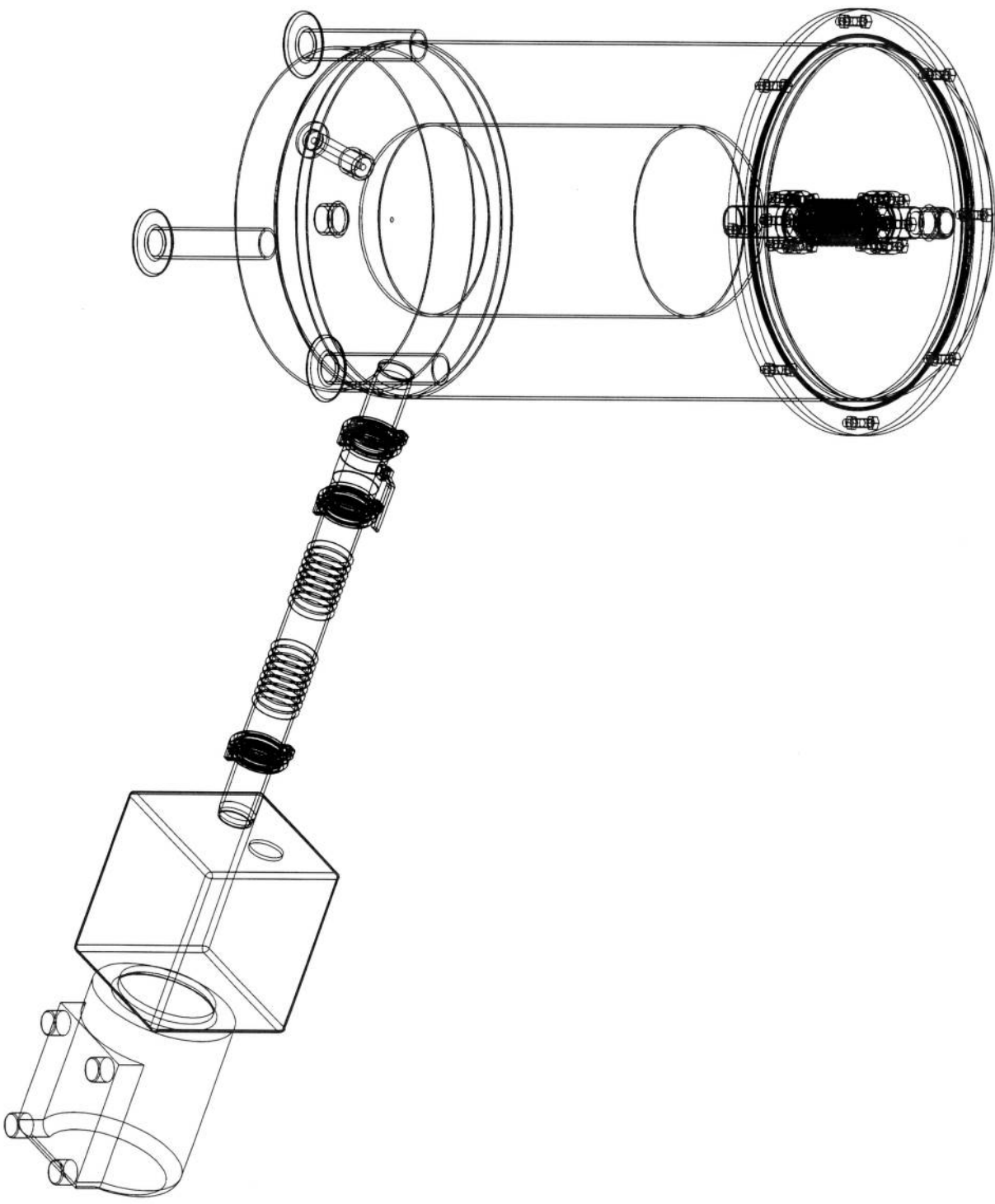
The following parts were obtained off the shelf.

Description	Supplier	Item No	Qty
DN 25 ISO-KF Corrugated Hose, Flexible	Air & Vacuum Technologies (Pty) Ltd	120SWN025-0500	1
DN 25 ISO-KF Centering Ring	Air & Vacuum Technologies (Pty) Ltd	112ZRG025	3
DN 25 ISO-KF Clamp	Air & Vacuum Technologies (Pty) Ltd	110BSR025	3
DN 25 Gate Valve	Air & Vacuum Technologies (Pty) Ltd	110VSM025	1
COF Elastomer Seal	Air & Vacuum Technologies (Pty) Ltd	602DOR300-S1	1
M6 Hexagon Head Screw with Nut	Air & Vacuum Technologies (Pty) Ltd	420BSC025-35	14
DN 25 CF Half Nipple Fixed Top / Bottom	Air & Vacuum Technologies (Pty) Ltd	420FRA025-25-52	2
DN 25 CF Copper Gasket	Air & Vacuum Technologies (Pty) Ltd	490DFL025-S10	20
ISO-KF Diaphragm Bellows	Air & Vacuum Technologies (Pty) Ltd	120SFM025-10	1

Appendix F: Design Drawings

The following manufacturing drawings were included for parts not obtained off the shelf.

DRAWING CONTENTS				
ASSEMBLIES	SUB-ASSEMBLIES	PARTS	ITEM NO.	DRAWING NO.
CRYOSTAT	CRYOSTAT EXTERNALS			CRYO
				CRYO - EX
		Outer Tank Wall	1	CRYO - EX 001
		Outer Tank Dome	2	CRYO - EX 002
		Stand	3	CRYO - EX 003
		DN 25 Vacuum Fitting	4	CRYO - EX 004
		DN 25 ISO-KF Corrugated Hose	5	-
		DN 25 ISO-KF Centering Ring	6	-
		DN 25 ISO-KF Clamp	7	-
		DN 25 Gate Valve	8	-
		E2M18 Edwards Vacuum Pump	9	-
		DN 16 Vacuum Coupling	10	CRYO - EX 005
		Outer Tank Flange	11	CRYO - EX 006
		COF Elastomer Seal	12	-
	CRYOSTAT INTERNALS	M6 Hexagon Head Screw	13	-
		M6 Hexagon Nut	14	-
				CRYO - IN
		DN 25 CF Half Nipple Fixed Top	1	-
		Coupling	2	CRYO - IN 001
		Improved Coupling A	20	CRYO - IN 0011
		Improved Coupling B	21	CRYO - IN 0012
		Optimum Coupling Design	22	CRYO - IN 0013
		Inner Tank Cylinder	3	CRYO - IN 002
		Inner Tank Top Dome	4	CRYO - IN 003
		Inner Tank Bottom Dome	5	CRYO - IN 004
		DN 25 CF Half Nipple Fixed Bottom	6	-
		M6 Hexagon Head Screw	7	-
		M6 Hexagon Nut	8	-
		DN 25 CF Copper Gasket	9	-
	MANUFACTURING	Male and Female Dies	-	CRYO - MA
Drawings of parts obtained from the shelf is not included - please check parts list and suppliers				



TITLE: CRYOSTAT

DIMENSIONS IN mm UNLESS OTHERWISE SPECIFIED
TOLERANCE: 0.1 UNLESS OTHERWISE SPECIFIED

MATERIAL: -

DWG NO. CRYO - 1

DRAWN: JH HODGMAN

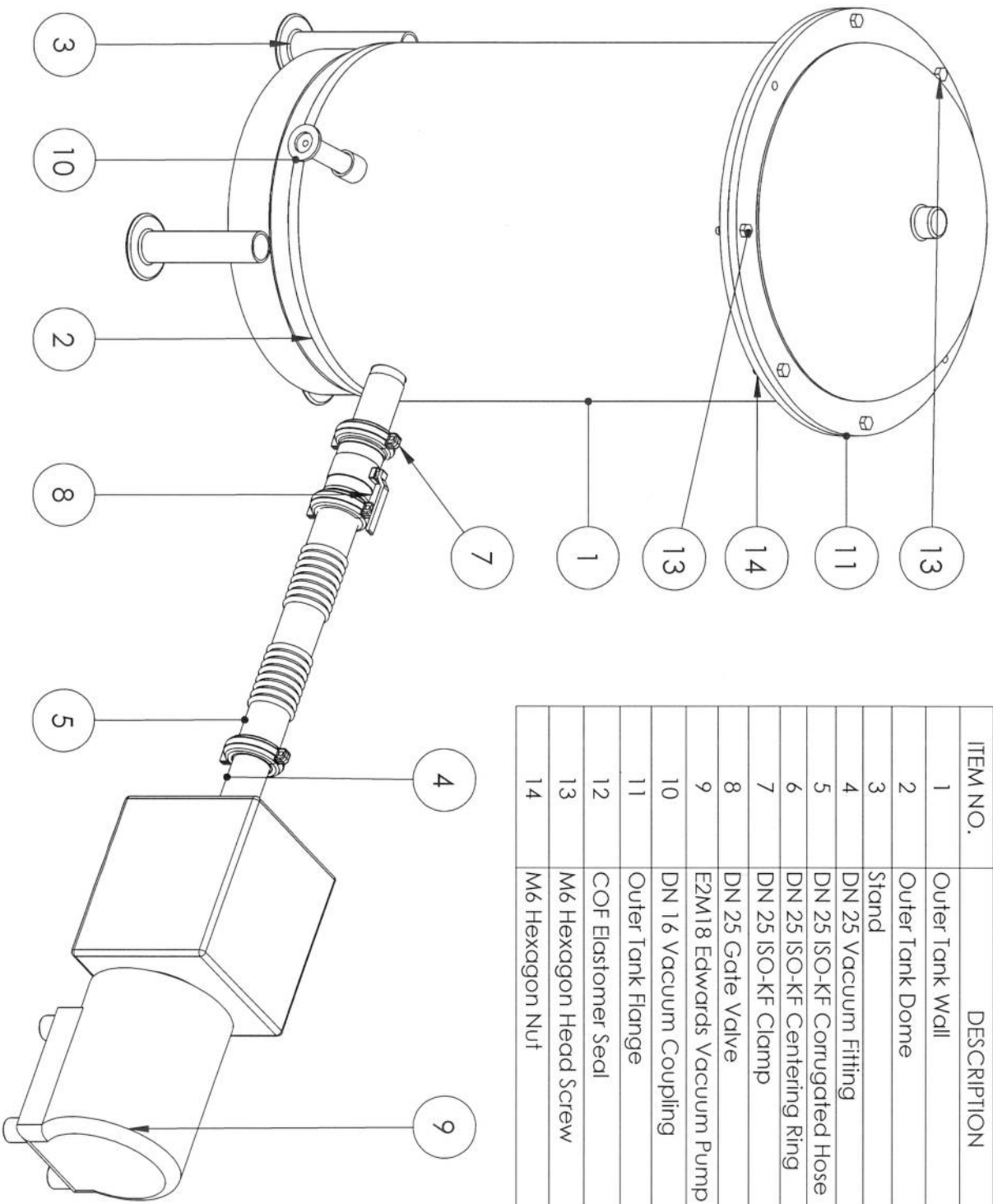
WEIGHT: 25 kg

SCALE: 1:5

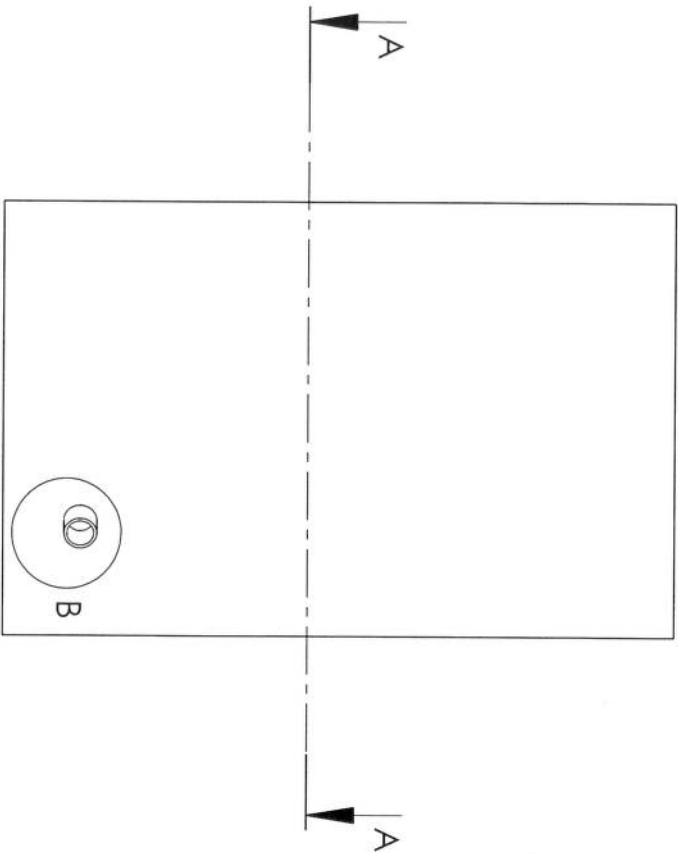
QTY: 1

A4

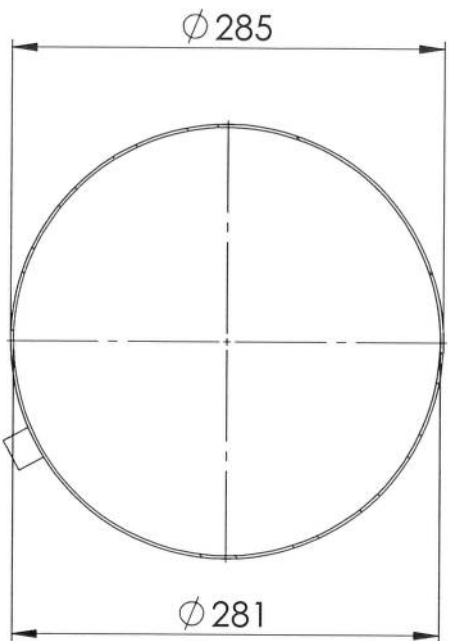
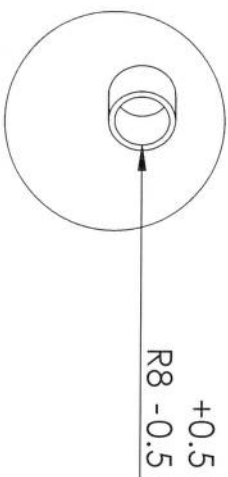
ITEM NO.	DESCRIPTION	DRAWING NO.	QTY.
1	Outer Tank Wall	CRYO - EX 001	1
2	Outer Tank Dome	CRYO - EX 002	2
3	Stand	CRYO - EX 003	1
4	DN 25 Vacuum Fitting	CRYO - EX 004	2
5	DN 25 ISO-KF Corrugated Hose	-	1
6	DN 25 ISO-KF Centering Ring	-	3
7	DN 25 ISO-KF Clamp	-	6
8	DN 25 Gate Valve	-	1
9	E2M18 Edwards Vacuum Pump	-	1
10	DN 16 Vacuum Coupling	CRYO - EX 005	1
11	Outer Tank Flange	CRYO - EX 006	2
12	COF Elastomer Seal	-	1
13	M6 Hexagon Head Screw	-	6
14	M6 Hexagon Nut	-	6



DIMENSIONS IN mm. UNLESS OTHERWISE SPECIFIED TOLERANCE: 0.5 UNLESS OTHERWISE SPECIFIED		TITLE: CRYOSTAT EXTERNALS		DEBUR AND BREAK SHARP EDGES	
MATERIAL: -	DWG NO. CRYO - EX	DRAWN: JH HODGMAN	A4		
WEIGHT: 16 kg	SCALE: 1:2	QTY: 1			



DETAIL B
SCALE 2 : 5



SECTION A-A
SCALE 1 : 5

DIMENSIONS IN mm UNLESS OTHERWISE SPECIFIED
TOLERANCE: 0.1 UNLESS OTHERWISE SPECIFIED

MATERIAL: SAE 1020

WEIGHT: 6.1 kg

TITLE: OUTER TANK WALL

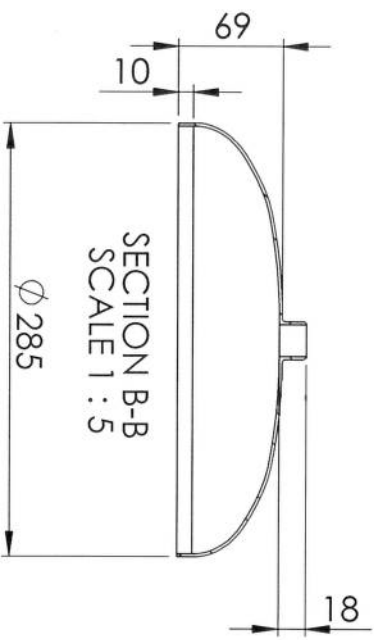
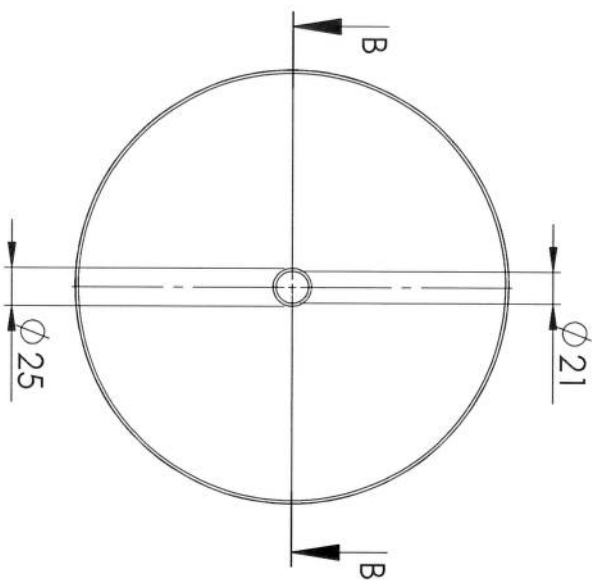
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SCALE: 1:5

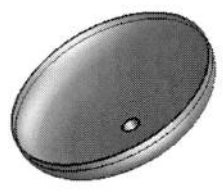
DRAWN: JH HODGMAN
DEBUR AND
BREAK SHARP
EDGES

QTY: 1

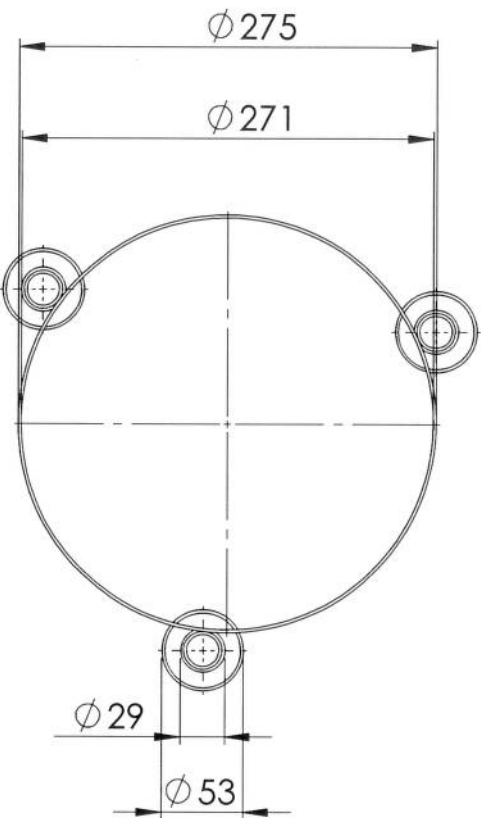
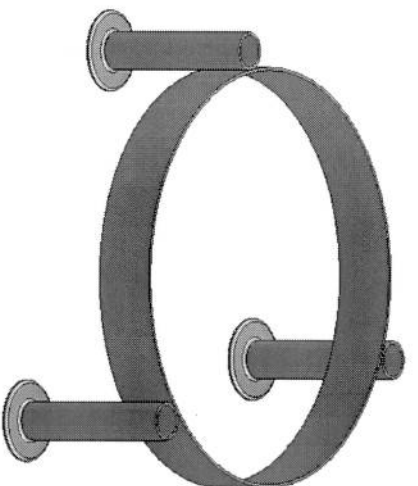
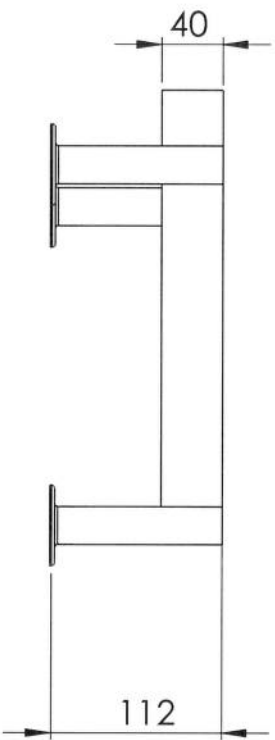
A4



SECTION B-B
SCALE 1 : 5



DIMENSIONS IN mm UNLESS OTHERWISE SPECIFIED TOLERANCE: 0.5 UNLESS OTHERWISE SPECIFIED		TITLE: OUTER TANK DOME		DEBUR AND BREAK SHARP EDGES	
MATERIAL: SAE 1020		DWG NO. CRYO - EX 002	DRAWN: JH HODGMAN	A4	
WEIGHT: 1.5kg		SCALE: 1:10	QTY: 2		



DIMENSIONS IN mm UNLESS OTHERWISE SPECIFIED
TOLERANCE: 1 mm UNLESS OTHERWISE SPECIFIED

MATERIAL: SAE 1020

WEIGHT: 1 kg

TITLE: STAND

DWG NO. CRYO - EX 003

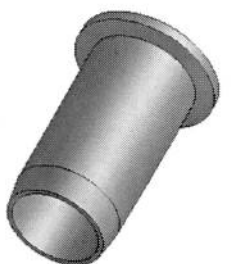
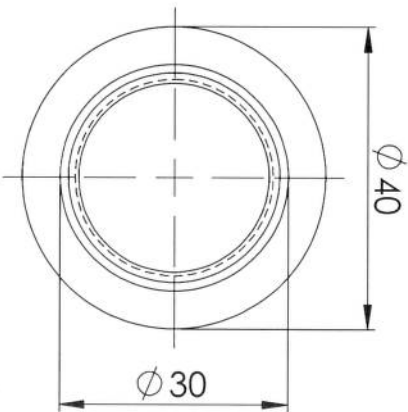
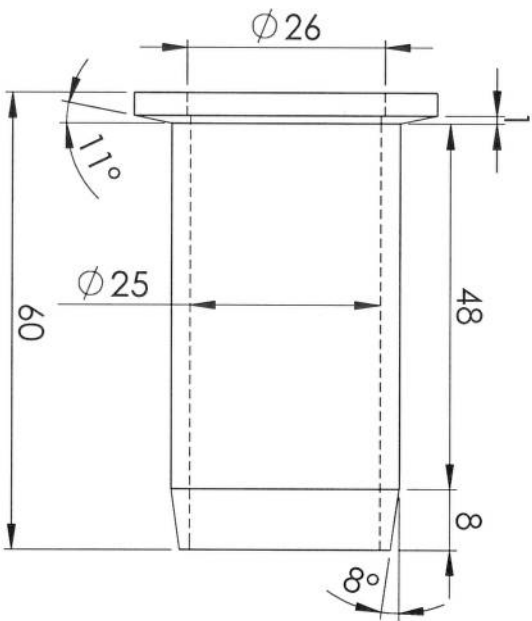
SCALE: 1:5

DEBUR AND
BREAK SHARP
EDGES

DRAWN: JH HODGMAN

QTY: 1

A4



DIMENSIONS IN mm UNLESS OTHERWISE SPECIFIED
TOLERANCE: 0.1 UNLESS OTHERWISE SPECIFIED

MATERIAL:
SAE 1020

WEIGHT: 0.1 kg

TITLE:
DN 25 Vacuum Fitting

DWG NO.
CRYO - EX 004

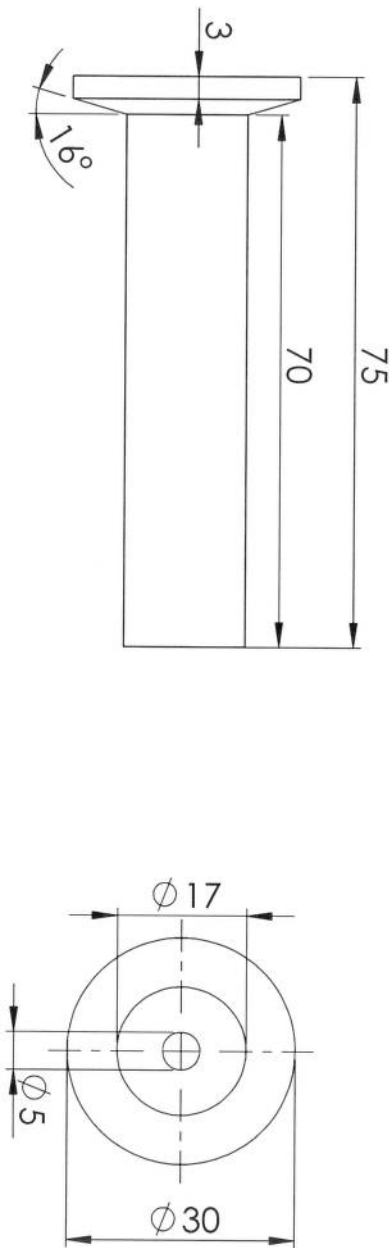
SCALE: 1:5

DEBUR AND
BREAK SHARP
EDGES

DRAWN:
JH HODGMAN

QTY: 1

A4



DIMENSIONS IN mm UNLESS OTHERWISE SPECIFIED
TOLERANCE: 0.1 UNLESS OTHERWISE SPECIFIED

MATERIAL: SAE 1020

WEIGHT: 0.1 kg

TITLE: DN 16 VACUUM COUPLING

DWG NO. CRYO - EX 005

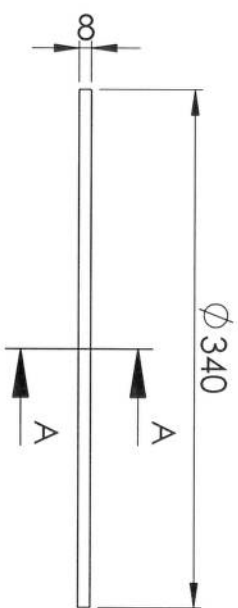
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DRAWN: JH HODGMAN

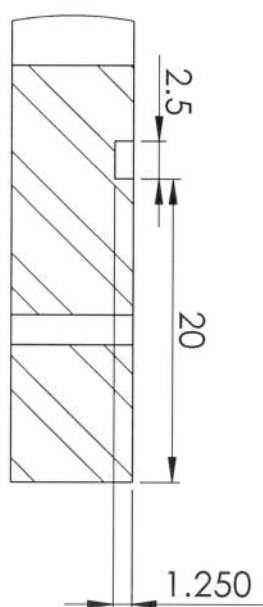
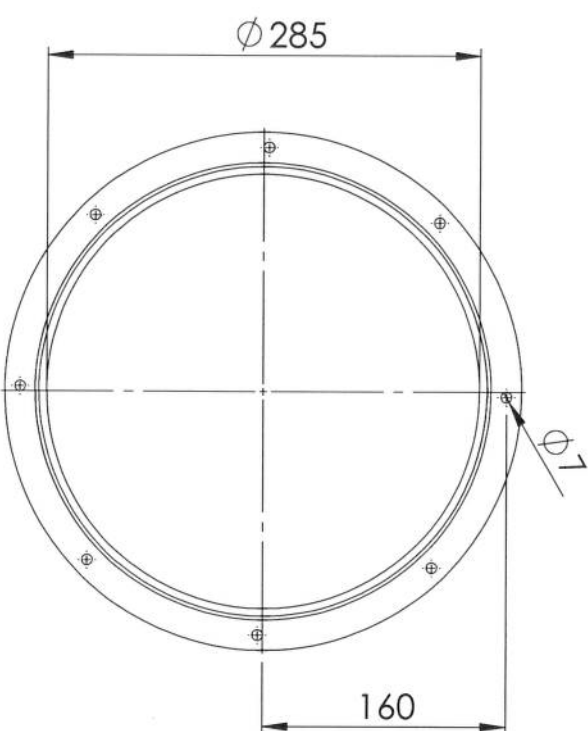
QTY: 1

A4

DEBUR AND
BREAK SHARP
EDGES



SECTION A-A
SCALE 1 : 5



DETAIL B
SCALE 2 : 1

DIMENSIONS IN mm UNLESS OTHERWISE SPECIFIED
TOLERANCE: 0.1 UNLESS OTHERWISE SPECIFIED

MATERIAL: SAE 1020

WEIGHT: 1.6 kg

OUTER TANK FLANGE

DWG NO. CRYO - EX 006

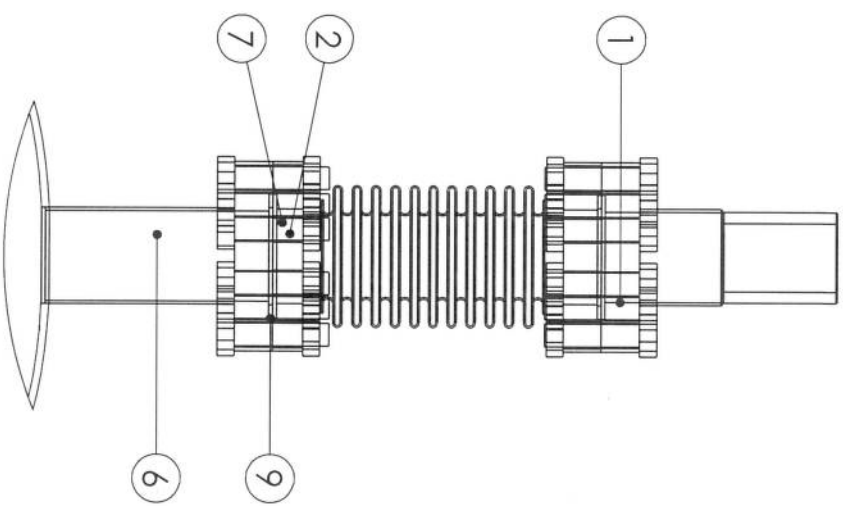
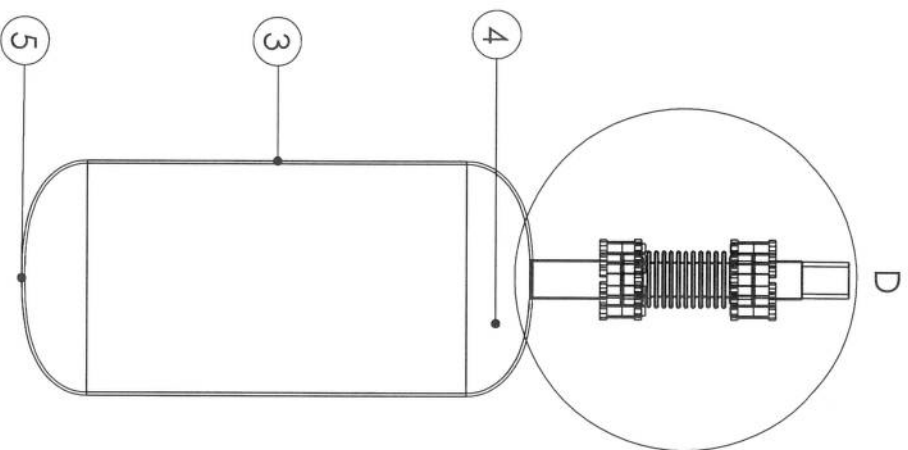
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DRAWN: JH HODGMAN

QTY: 2

DEBUR AND
BREAK SHARP
EDGES

A4

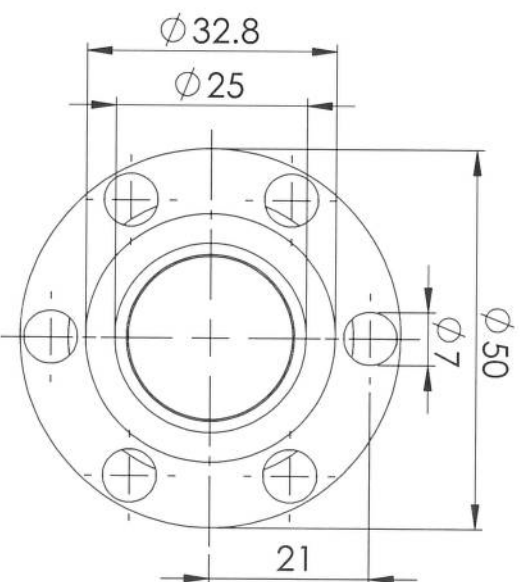
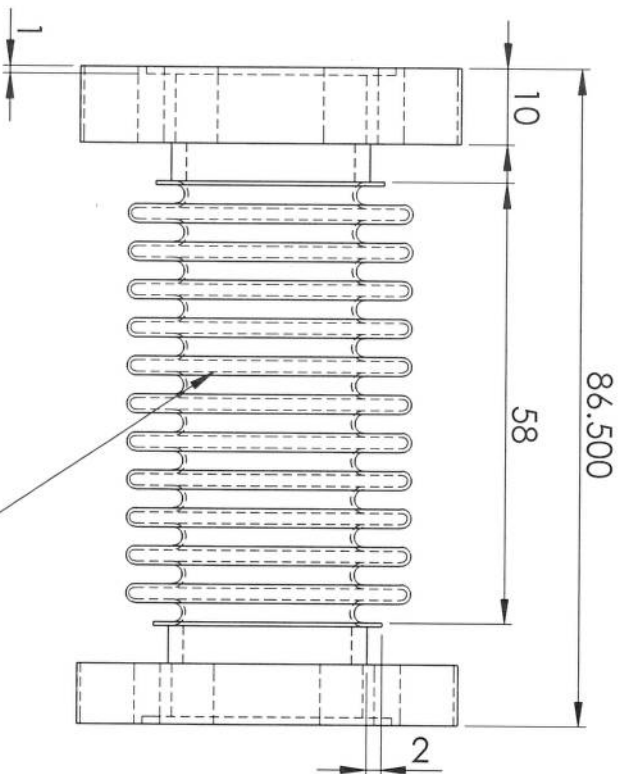


ITEM NO.	DESCRIPTION	DRAWING NO.	QTY.
1	DN 25 CF Half Nipple Fixed Top	CRYO - IN 001	1
2	Coupling	CRYO - IN 002	1
3	Inner Tank Cylinder	CRYO - IN 003	1
4	Inner Tank Top Dome	CRYO - IN 004	1
5	Inner Tank Bottom Dome	-	1
6	DN 25 CF Half Nipple Fixed Bottom	-	12
7	M6 Hexagon Head Screw	-	12
8	M6 Hexagon Nut	-	12
9	DN 25 CF Copper Gasket	-	2

DIMENSIONS IN mmS UNLESS OTHERWISE SPECIFIED TOLERANCE: 0.1 UNLESS OTHERWISE SPECIFIED		TITLE: CRYOSTAT INTERNALS		DEBUR AND BREAK SHARP EDGES	
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MATERIAL: -	DWG NO. CRYO - IN	DRAWN: JH HODGMAN	A4
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WEIGHT: 9 kg	SCALE: 1:2	QTY: 1
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ISO - KF Spring Bellow 0.3 mm wall thickness

DIMENSIONS IN mm UNLESS OTHERWISE SPECIFIED
TOLERANCE: 0.1 UNLESS OTHERWISE SPECIFIED

MATERIAL: SAE 304 L

TITLE: COUPLING

DWG NO. CRYO - IN 001

DEBUR AND
BREAK SHARP
EDGES

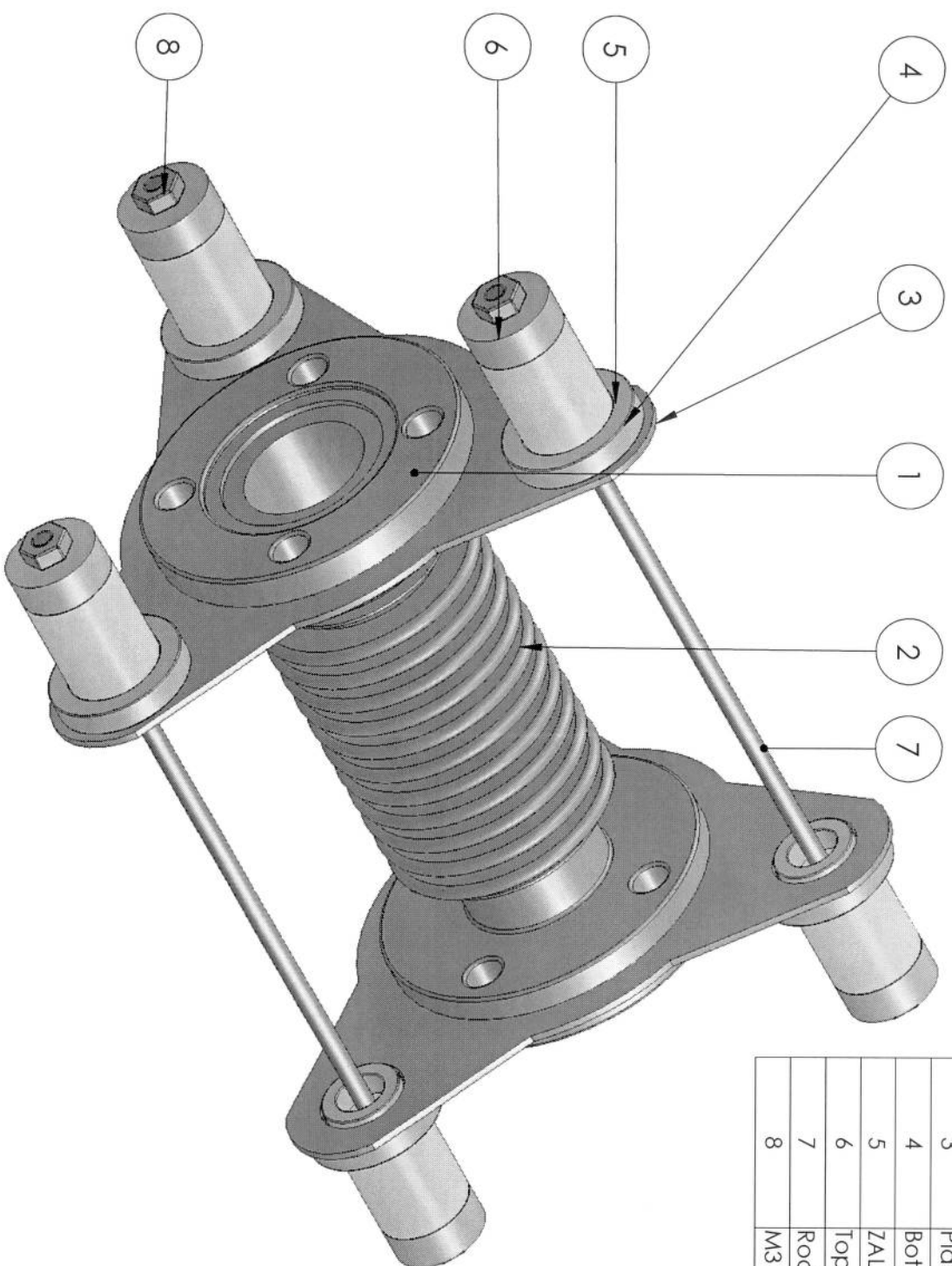
DRAWN: JH HODGMAN

A4

WEIGHT: 0.25 kg

SCALE: 1:1

QTY: 1



ITEM NO.	DESCRIPTION	QTY.
1	CF Half Nipple Fixed	2
2	Bellowed Hose	1
3	Plate Support	2
4	Bottom Spacer	6
5	ZAL 45 Insulation	6
6	Top Spacer	6
7	Rod	3
8	M3 Hexagon Nut	6

DIMENSIONS IN mmS UNLESS OTHERWISE SPECIFIED
TOLERANCE: 0.5 UNLESS OTHERWISE SPECIFIED

MATERIAL: -

WEIGHT: 0.4 kg

TITLE: IMPROVED COUPLING A

DEBUR AND
BREAK SHARP
EDGES

DWG NO.

CRYO - IN 0011

DRAWN:

JH HODGMAN

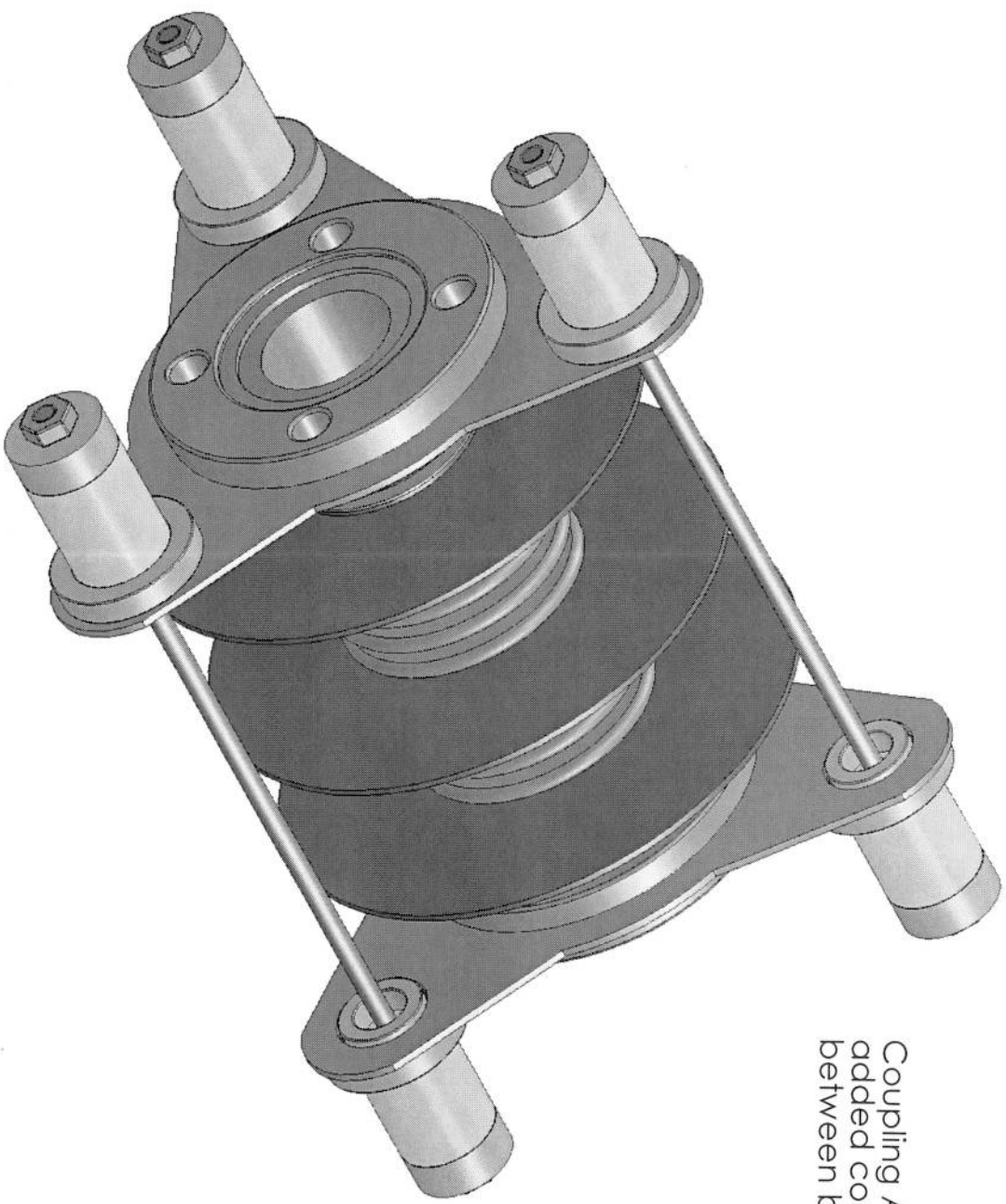
A4

SCALE: 1:2

QTY:

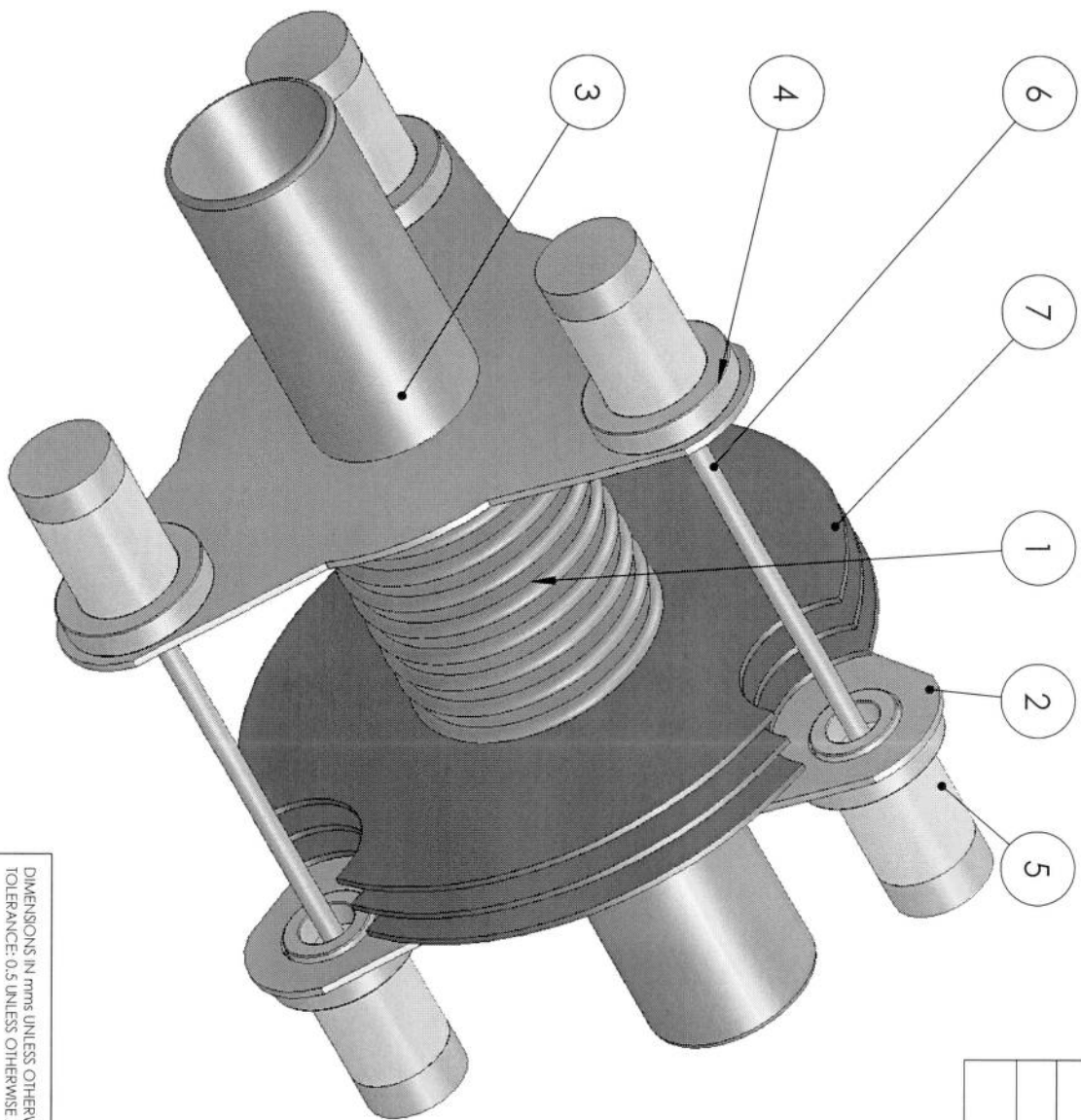
1

Coupling A with
added copper heat sinks
between bellowed pipe vanes.



DIMENSIONS IN mmS UNLESS OTHERWISE SPECIFIED TOLERANCE: 0.5 UNLESS OTHERWISE SPECIFIED		TITLE: IMPROVED COUPLING B		DEBUR AND BREAK SHARP EDGES		
MATERIAL: -	DWG NO. CRYO - IN 0012	DRAWN: JH HODGMAN				A4
WEIGHT: 0.4 kg	SCALE: 1:2	QTY: 1				

ITEM NO.	DESCRIPTION	MATERIAL	QTY.
1	Bellowed Hose	SAE 304L	1
2	Plate Support	Fibre Glass	2
3	Inlet Pipe	SAE 304L	2
4	Bottom Spacer	Aluminium	6
5	ZAL 45 Insulation	ZAL 45 Board	6
6	Top Spacer	Aluminium	3
7	Copper Heat Sink	Copper	3



DIMENSIONS IN mmS UNLESS OTHERWISE SPECIFIED
TOLERANCE: 0.5 UNLESS OTHERWISE SPECIFIED

MATERIAL: -

WEIGHT: 0.4 kg

TITLE:

OPTIMUM COUPLING DESIGN

DEBUR AND
BREAK SHARP
EDGES

DWG NO.

CRYO - IN 0013

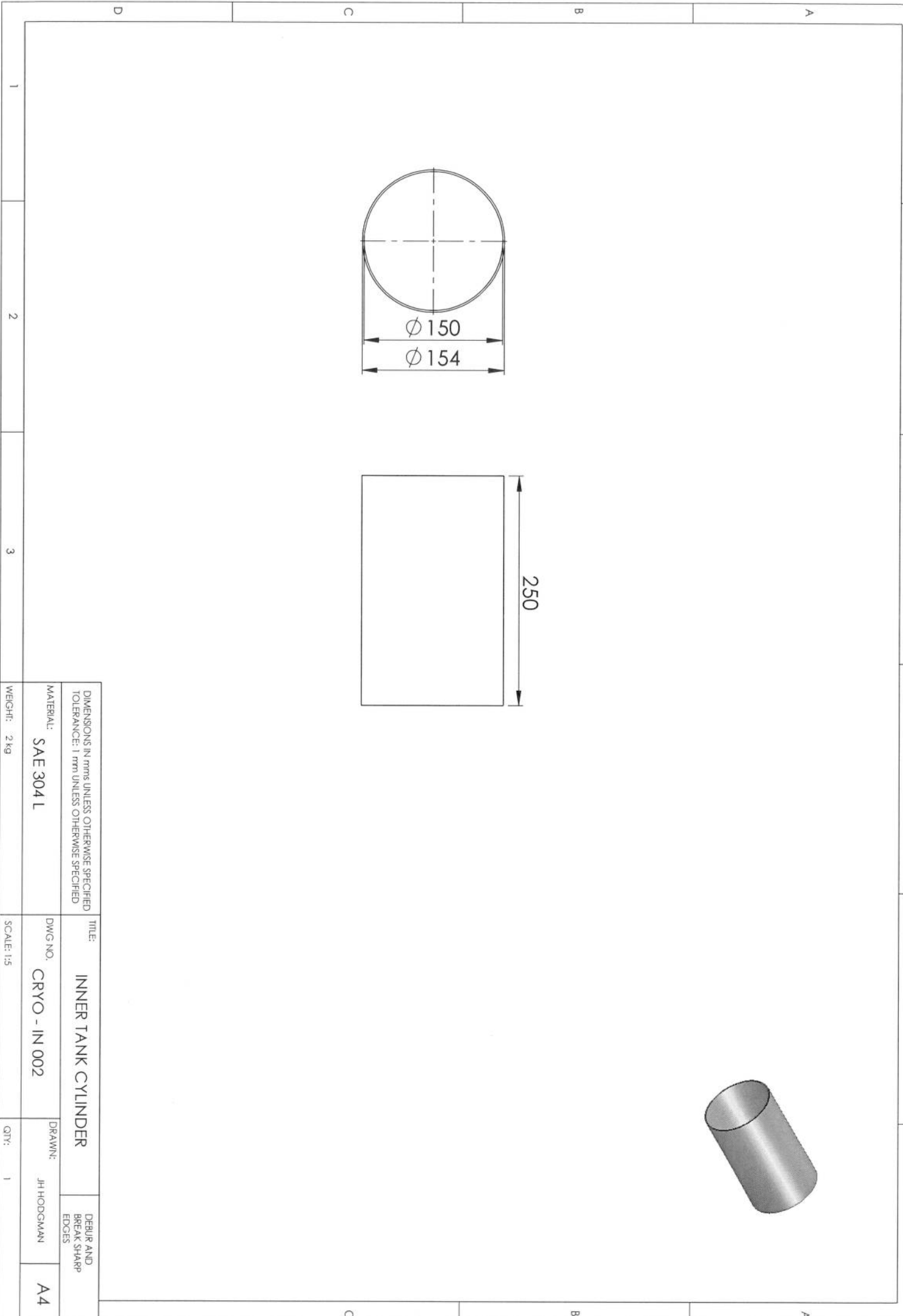
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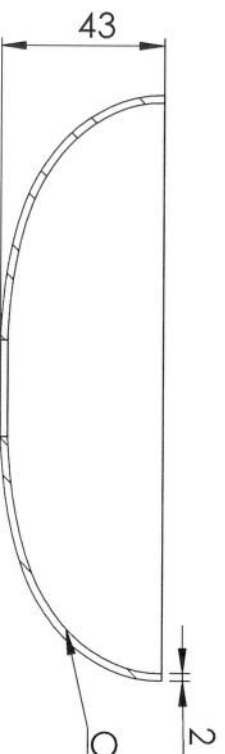
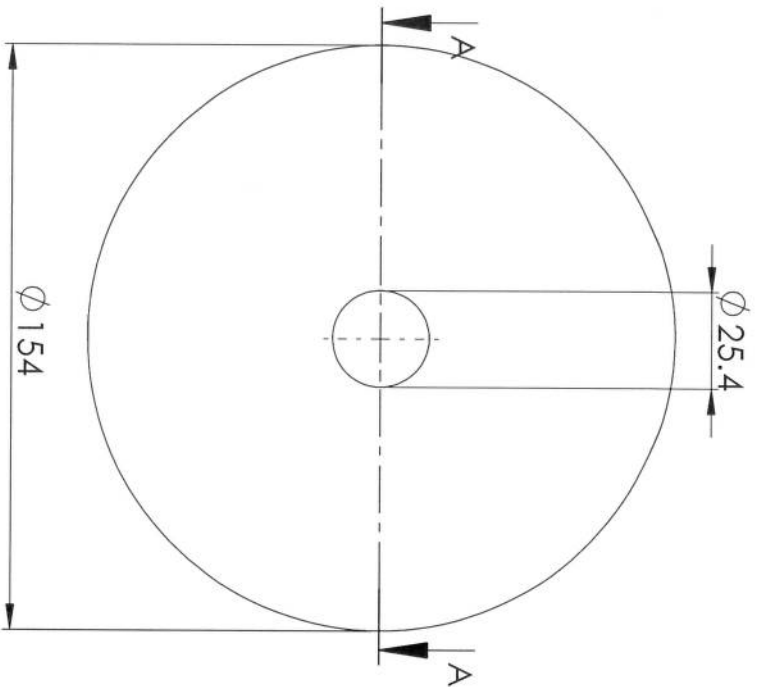
JH HODGMAN

A4

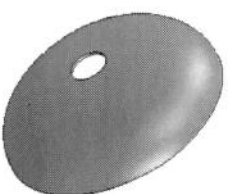
SCALE: 1:2

QTY: 1





SECTION A-A
SCALE 1 : 2



DIMENSIONS IN mm UNLESS OTHERWISE SPECIFIED
TOLERANCE: 0.5 UNLESS OTHERWISE SPECIFIED

MATERIAL: SAE 304 L

WEIGHT: 0.4 kg

TITLE: INNER TANK TOP DOME

DWG NO. CRYO - IN 003

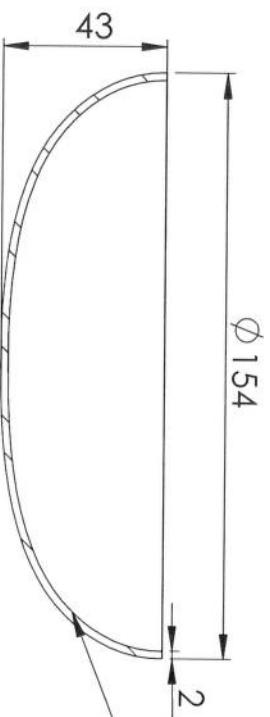
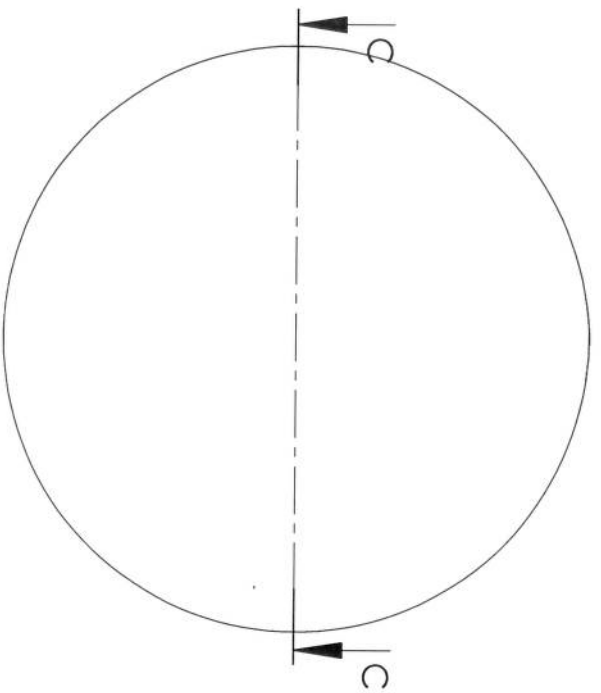
SCALE: 1:2

DEBUR AND
BREAK SHARP
EDGES

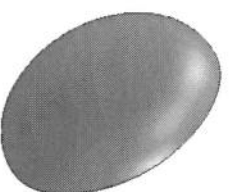
DRAWN: JH HODGMAN

QTY: 1

A4



SECTION C-C
SCALE 1 : 2



DIMENSIONS IN mm UNLESS OTHERWISE SPECIFIED
TOLERANCE: 0.5 UNLESS OTHERWISE SPECIFIED

MATERIAL: SAE 304 L

WEIGHT: 0.4 kg

TITLE: INNER TANK BOTTOM DOME

DWG NO. CRYO - IN 003

SCALE: 1:2

DEBUR AND
BREAK SHARP
EDGES

DRAWN: JH HODGMAN

QTY: 1

A4