

“Design of a Laboratory Nitrogen Liquefier cum Cryostat Based on Closed Cycle Refrigerator”

*A dissertation submitted in partial fulfilment of the requirement
FOR THE DEGREE OF*

**MASTER OF SCIENCE
IN PHYSICS**

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CERTIFICATE

This to certify that, the work in the thesis entitled ***“Design of a Laboratory Nitrogen Liquefier cum Cryostat Based on Closed Cycle Refrigerator”*** by **Ganga Thapa**, in partial fulfilment of the requirements for the award of degree of Master of Science in Physics at the National Institute of Technology, Rourkela is an authentic work carried out under my supervision and guidance.

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ACKNOWLEDGEMENT

I sincerely record my deep gratitude to my project guide Dr. Prakash Nath Vishwakarma for his wonderful supervision & constant encouragement to carry out this work.

I gratefully acknowledge the help of Mr. Balaji Choudhary, Research Scholar, Mechanical Department in the completion of this project.

I also take this opportunity to thank my friend Soumyakanta Swain, B.Tech, Mechanical Department for making me familiar with engineering outlook.

I also express my indebtedness towards all mentioned in the reference section. This kind of work is never possible without them.

And last but not the least I shall remain thankful to my seniors & friends for their support & affection all this while.

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ABSTRACT

Taking the CRYOMECH LNP 40 specifications as a reference, an attempt was made to design an integrated system consisting of a liquefying unit with a measurement cryostat. The dimensions were calculated in a very crude way though always taking the upper value. The dimensions for different parts were set with special care given to the sample chamber. Then a sample solid model was made. Heat transfer calculations were done to estimate the total heat going into the system from the major possible sources. Staying time of the cryogen was calculated. Encouragingly a reasonable value was obtained. And it was found out that with efficient cooling capacity liquefier, the system can be very handy both for measurement as well as transferring.

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1 – INTRODUCTION

1.1 Cryogenics, A Historical Review:

The temperature of any material- solid, liquid or gas- is a measure of the energy it contains. If 'T (in Kelvin scale)' is the temperature then the corresponding energy is given by $E=k_B T$, k_B being the Boltzmann constant. This energy is due to various forms of motion among the atoms or molecules of which the material is made. A gas that consists of very rapidly moving molecules, for example, has a higher temperature than one with molecules that are moving more slowly. So manipulation of temperature means exploitation of material properties. This is the essence of Low Temperature Physics more sophisticatedly abbreviated as "Cryogenics."

Cryogenics deals with the study of production of very low temperature & behaviour of materials at those temperatures. The word cryogenics comes from the Greek word cryos, meaning "cold," combined with a shortened form of the English verb "to generate". It means, generation of temperatures well below those of normal human experience. More specifically, a low-temperature environment is termed a cryogenic environment when the temperature range is below the point at which all the gases (such as oxygen, nitrogen, hydrogen, helium, etc) begin to liquefy.

The origin of cryogenics as a scientific discipline coincided with the discovery that the permanent gases can also be liquefied at exceedingly low temperatures. Consequently, the term "cryogenic" applies to temperatures from approximately 123K (−150°C) down to absolute zero (the coldest point a material could reach, but it never gets there!). But it should be always kept in mind that there is no precise demarcation of temperature in this respect.

It is actually one of those few branches of science where mankind has surpassed nature. The lowest temperature in the universe is 2.73K. But in laboratory the temperature of the order of micro Kelvins has been achieved (by Nuclear Demagnetisation). This is a great enthralling element for the people associated with this field.

1.2 Liquefaction of Gases:

Liquefaction of gases provides the simplest route to low temperatures & this continues to be the most desirable method till now. Anything may be cooled simply by contact with the liquid. That contact can be direct or indirect depending on the cooling technique.

Liquefaction of gases is physical conversion of a gas into a liquid state (condensation). It is the process of refrigerating a gas to a temperature below its critical temperature so that liquid can be formed at some suitable pressure, also below the critical pressure. Below the critical temperature the gas is termed as vapour.

For a long time it was thought that air remains in the gaseous state at all temperatures. Therefore gases like oxygen, nitrogen, helium etc. were termed as permanent gases. In 1823 it was Faraday who first found out traces of liquid chlorine in a sealed glass tube produced by the pressure built up in the closed tube. This is how it began.

The nineteenth century was the exciting period for gas liquefaction. There was a race to liquefy these so called permanent gases. Below is a chronological account of this pedagogical pursuit:

- In 1823 Faraday at Royal Institution in London liquefied chlorine by heating & consequent decomposing in a sealed glass tube.
- In 1877 Cailletet produced a 'mist' of oxygen by compressing the gas in certain conditions & then suddenly releasing it. But six years later, it was von Wroblewski &

Olszewski who actually liquefied it with some improved conditions. They did it successfully for CO₂ & N₂. It was them who first used a second chamber outside the chamber containing the cryogen.

- In 1898 Sir James Dewar liquefied hydrogen using Joule-Thomson cooling. And the dewar vessel was also invented along with it. So it was a very important step in liquefaction & then its storage as well.
- In 1908 H. K. Onnes at Leiden liquefied helium (⁴He), not intentionally though. He was trying to build large capacity air & hydrogen liquefiers. This opened up a vast realm of research for the scientists. And most of the pioneering low temperature research work has been carried out in the University of Leiden. In fact H. K. Onnes is considered to be “The Father of the Low-Temperature Physics”.

1.3 Properties of Cryogen:

The equation of state for an ideal gas is given by,

$$PV = nRT$$

where P is the pressure, V is the volume & n is the number of moles of gas. T is the temperature of gas in Kelvin & R being the universal gas constant with value 8.314 Joule/mole K.

But for a real gas, the modified eqⁿ with Vander Waal's correction is,

$$(P + a/V^2) (V-b) = n RT$$

where the term a/V^2 accounts for the change in pressure due to the intermolecular forces & b is the decrease in volume due to finite size of the gas molecules.

The plot obtained between P vs. V at a constant temperature is known as isotherm. Given below (Fig. 1.1) is a plot of isotherms for real gas.

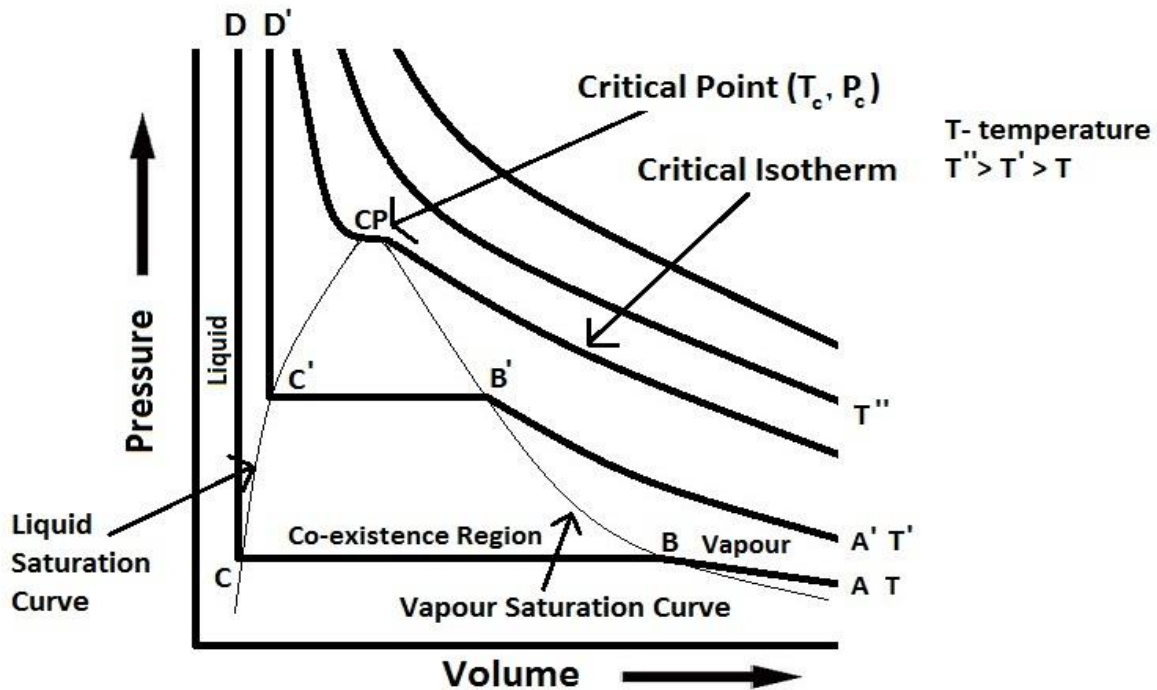


Fig. 1.1: Plot of isotherm at different temperatures for a gas. The dotted curve marks the boundary of the 2-phase coexistence region. The critical point is indicated.

The isotherms give you an insight into the behaviour of gases at different temperature. The curve, at high temperature obeys the ideal gas eqⁿ $PV = \text{constant}$. But at the lower temperature, we encounter a temperature below which the gas liquefies. This is the critical temperature T_c which corresponds to a critical pressure P_c .

Along the region AB & $A'B'$, the gas compresses so that it begins to liquefy below the point B & B' respectively. And further compression leads to increase in liquefaction at the same pressure. After the point C , there's a sudden rise in the pressure of the liquid. So this implies that at the point C , all the gas has been liquefied. The curve $CC'CP$ is called the liquid saturation curve. And similarly the curve $BB'CP$ curve is known as the vapour saturation

curve. BC & B'C' regions represent the co-existence of the liquid & vapour phase having temperature equal to the boiling point of the liquid.

The critical temperature happens to be the one at which the co-existence between the two phase vanishes. In the region BC & B'C', the heat is utilised in conversion from liquid phase to vapour phase. This is nothing but the latent heat of vaporisation L_v .

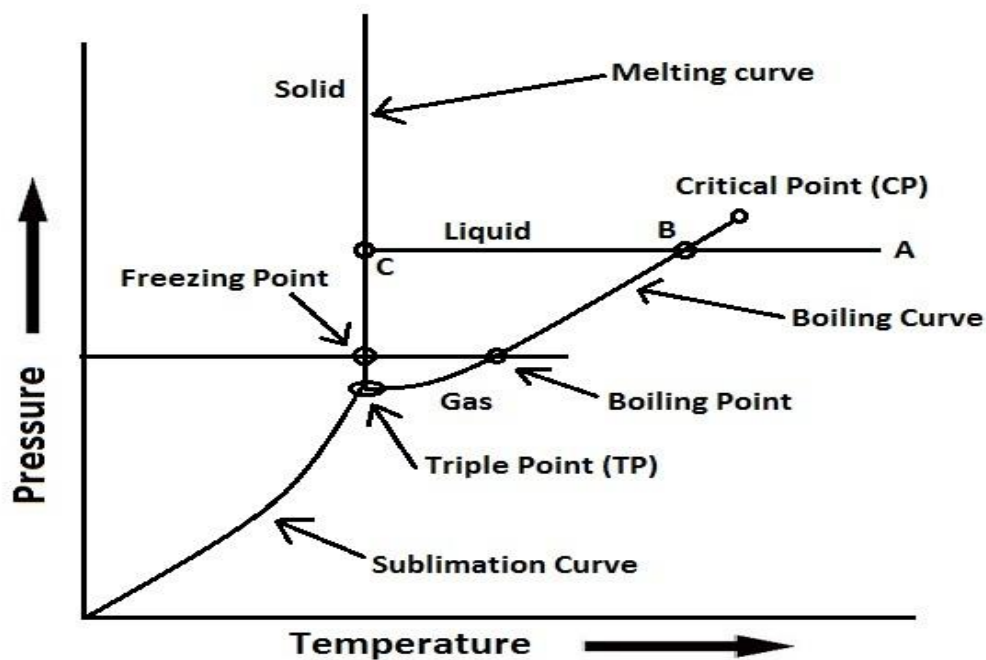


Fig. 1.2: Boiling, melting & sublimation curves for a fluid

Now fig.1.2 is a plot of temperature as a function of pressure. It's a typical phase diagram for a fluid.

Triple point is a single combination of pressure & temperature at which the three phases of a substance co-exist in thermodynamic equilibrium. The boiling & melting point of a substance meet at the triple point.

Suppose we lower the temperature of a gas at a constant pressure from the point A. From the point B, it starts liquefying. If the temperature is lowered further, then after reaching the point C, it begins to freeze. The plot thus has three distinct regions as shown.

Below the triple point the gas goes into the solid phase. So this has to be the lowest temperature that we can have in a cryogenic bath. And of course the upper limit is given by the critical temperature.

Except Helium all gases have a triple point (Fig. 1.3). Helium has 2 isotopes with mass number 4 & 3. ^4He has an isotopic abundance of 99.7%. Normally the word 'Helium' refers ^4He .

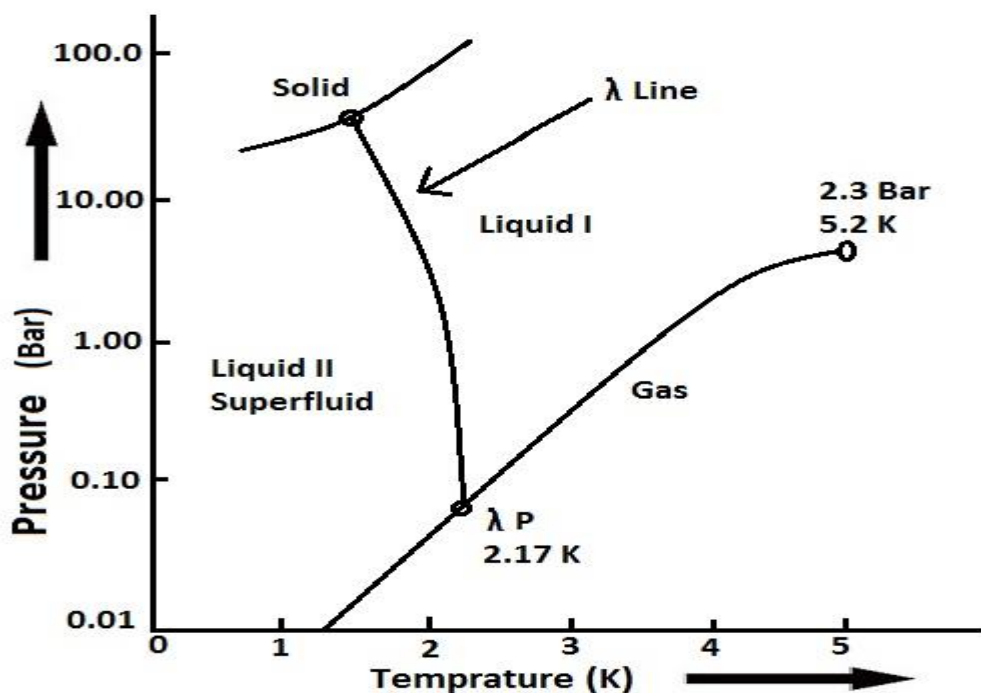


Fig. 1.3: Phase Diagram for ^4He . The solid, liquid, & vapour phases in ^4He do not coexist. However Liquid I (normal), Liquid II (superfluid) & solid phases coexist.

At a temperature of 2.17K & pressure $\approx 50\text{mbar}$, there's a drastic change in the properties of liquid Helium. It transforms to the superfluid phase where its viscosity ceases to exist. The curve resembles the Greek symbol lambda, hence the transition is known as lambda transition. The lambda transition line meets the boiling line of liquid helium at a point λP called Lambda Point. Now this can be called a triple point for Helium at which the phase I, phase II & gas phase are in equilibrium.

The table below gives an account of physical data for gases. And the corresponding data for water is also listed for ready comparison.

Fluid	T _b (K) at 1 bar	T _c (K)	P _c (bar)	T _{TP} (K)	P _{TP} (mbar)	L(kJ/litre) at T _b
⁴ He	4.21	5.2	2.28	-	-	2.56
³ He	3.19	3.32	1.16	-	-	0.48
N ₂	77.4	126.0	33.9	63.15	0.13	160
Ar	87.3	150.9	48.7	83.81	0.67	224
O ₂	90.2	154.3	50.4	54.36	0.015	245
H ₂ O	373.15	647.13	220	273.16	0.06	2252

Table 1

1.4 Closed Cycle Refrigerator (CCR):

Closed Cycle Refrigerator (CCR) made the low temperature accessible for widespread use. It is a standalone refrigerator which produces low temperature continuously by repeating the cooling process in cycles. The cooling circuit is a closed one. Hence the name- closed cycle refrigerator. Cyclic refrigeration is useful because it gives us continuous production of low temperature. A CCR has following components:

- ❖ A compressor
- ❖ A displacer
- ❖ Heat exchanger
- ❖ Regenerator

I. Compressor:

A compressor is a mechanical device that increases the pressure of a gas by reducing its volume. These are similar to pumps: both increase the pressure on a fluid and both can transport the fluid through a pipe. They collect and store air in a pressurized tank, and use pistons and valves to achieve the appropriate pressure levels within an air storage tank that is attached to the motorized unit.

II. Displacer:

It moves the gas from the cold to warm end and vice-versa.

III. Heat exchanger:

A heat exchanger is a piece of equipment built for efficient heat transfer from one medium to another. The media may be separated by a solid wall, so that they never mix, or they may be in direct contact.

Usually in a liquefier the pressure on the liquefied gas will be close to the atmospheric pressure. Since the normal boiling point of all these gases is considerably below room temperature, the gas must undergo progressive cooling over a period of time for it to be liquefied. This is done by using a heat exchanger.

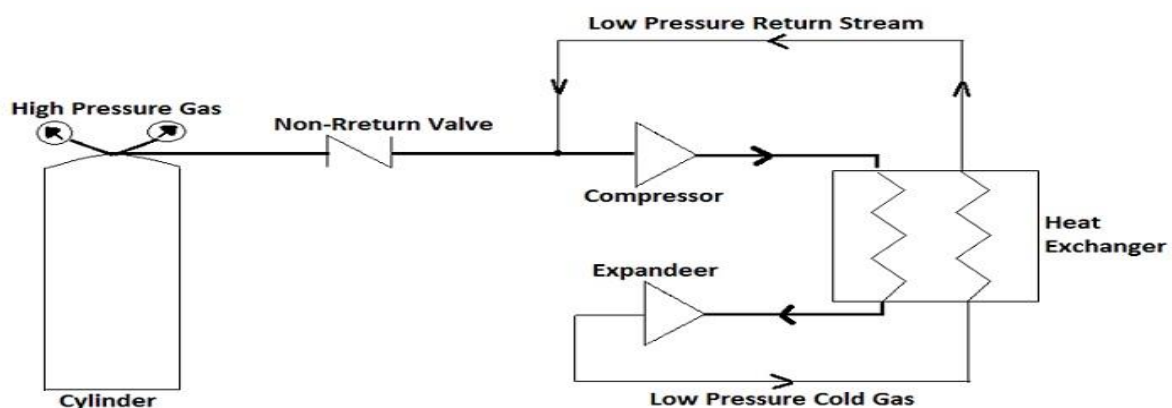


Fig. 1.4: Iterative cooling using a recuperative heat exchanger

The principle of iterative cooling is illustrated in fig. 1.4. There are two paths for the flow of the gas. The high pressure stream flows through one path & the low pressure stream flows in the opposite direction in the other path. To obtain iterative cooling compressed gas at room temperature flows through the high pressure arm of the heat exchanger. A part or the whole of the gas undergoes expansion which produces cold gas at low pressure. This cold gas then passes through the low-pressure path in the heat exchanger in counter flow. It cools the incoming high-pressure stream & enters as room temperature gas at the inlet of the compressor. It is compressed again & undergoes this cyclic operation. The gas coming out of the high pressure path of the heat exchanger gets cooled progressively & reaches a temperature much below the inversion temperature.

IV. Regenerator:

A **Regenerative heat exchanger**, or more commonly a **regenerator**, is a type of heat exchanger where the flow through the heat exchanger is cyclical and periodically changes direction. At any instant of time there is only one stream of gas, either warm or cold. It serves as a repository of heat from the warm gas when it passes through the regenerator & as a supplier of heat when the cold gas passes through it.

➤ Two of the widely used CCRs are:

❖ Gifford-McMohan Refrigerator

❖ Pulse Tube Refrigerator

(A) Gifford-McMohan Refrigerator:

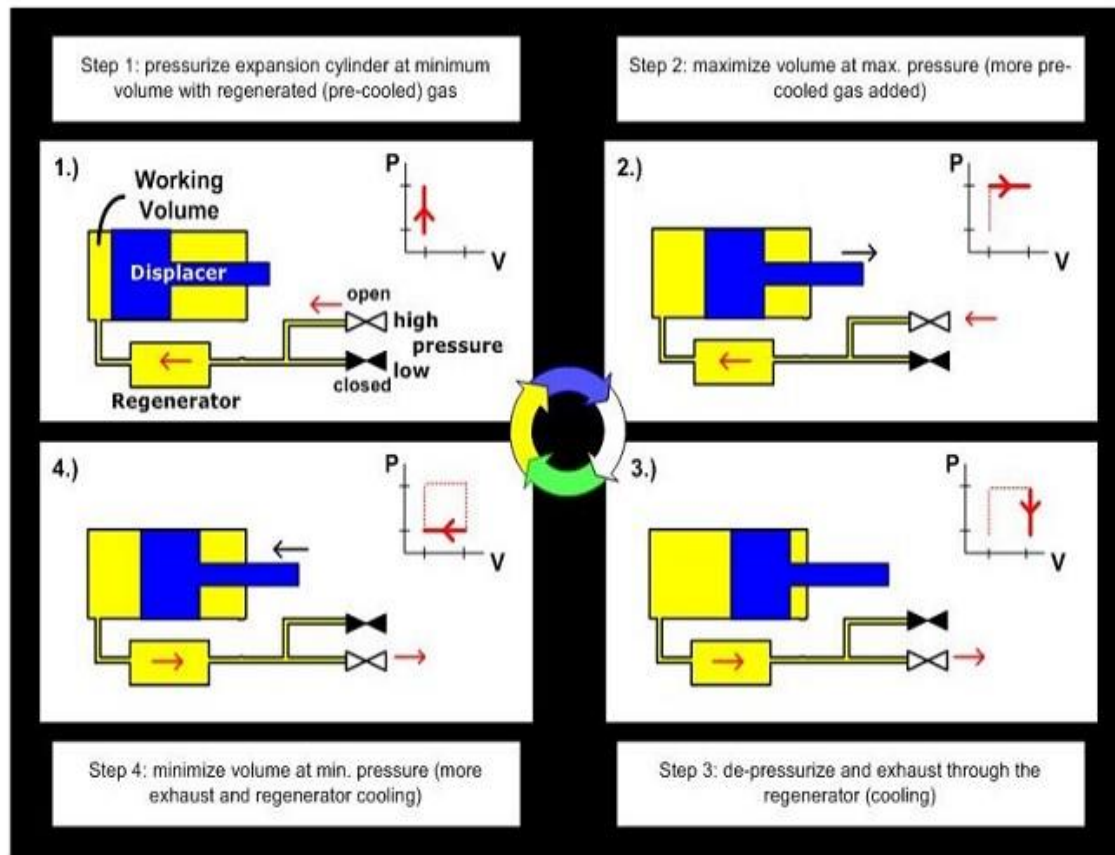


Fig.1. 5: Operation of GM Cycle

The basic operation of the GM cycle is illustrated in the above fig. 1.5.

It consists of four phases. They are as follows:

- (i) In the 1st phase called as the pressurization phase, the compressed gas is allowed to flow into the cylinder. The cold volume is at the minimum possible volume now.
- (ii) Then the intake phase comes into play. During this the intake valve (at high pressure) still remains open. The displacer is released to move upward. This causes the warm gas to pass through the regenerator.
- (iii) Now the intake valve is closed & the expansion phase begins. The exhaust valve (at low pressure) is opened. The expansion leads to cooling.

(iv) The end phase is the exhaust phase. The displacer is moved downwards to push out the cold gas through the regenerator. Then the exhaust valve is closed & the cycle is repeated.

GM coolers are very much in demand these days for communication electronics, cryopumping & MRI installations. It is very widely used because one can easily multi-stage it to get down to lower temperature.

(B) Pulse Tube Refrigerator:



Fig. 1.6: A Pulse Tube Refrigerator

This eliminates the moving components of the GM refrigerator replacing it with the gas piston which makes it more reliable. The reservoir volume is sufficient to reduce any pressure oscillation during the flow. The oscillating flow through the orifice separates the heating & cooling effects.

The different parts of a Pulse Tube refrigerator are as given below:

- **Hot heat exchanger:** Release the heat created in the compression cycle to the environment.
- **Cold heat exchanger:** Absorbs the heat of the environment because of cooling down in the expansion cycle.
- **After cooler (AC):** Removes the heat of the compression in the compressor.

- **Buffer:** A reservoir having much more volume in compare with the rest of the system practically at constant pressure.
- **Orifice:** An inlet for the flow resistance.
- **Compressor:** Creating a harmonic oscillation for the gas inside the system. Comes with a piston that moves back & forth.

Basic Operation:

The region between the two heat exchangers is isolated from the environment. Pressure increases & decreases as the gas moves from the right to left driven by the piston's movement. When the compressed gas moves towards the right, the tube gets heated. Then when the pressure is decreased by the flow from the reservoir into the tube via the orifice, adiabatic expansion takes place leading to cooling effect. An asymmetrical heating effect is produced where one end becomes hot & the other end keeps getting cooler.

In the repeated compression & expansion, the heat exchangers maintain a steady temperature to substantiate this effect. The after cooler causes the gas to comeback to ambient temperature. And the regenerator takes up the heat from the cold gas. The next batch of incoming gas takes in the cold stored in the regenerator. Hence the temperature of the gas entering this time is less than the previous cycle. And with repeated cycle the desired cooling occurs. It's evident that the cooling depends on the heat load entering the system.

1.5 Cryostat:

It's a device where low temperature is maintained for specific purposes like measurement at low temperature or frozen section histology of biological sample. According to how that low temperature is obtained, it is of following types:

(i) Bath Cryostat:

In this the entire experimental set up is immersed in the cryogenic bath & is suspended from a top flange. It would be similar to a storage dewar in construction. The liquid cryogen may be replenished once it boils away, at specified intervals depending on the volume and construction of the cryostat.

(ii) Continuous Flow Cryostat:

In this type, the liquid cryogen is sucked by a vacuum pump & flows through the different parts of the system to be cooled. This is particularly useful when the experiments have to be done at variable low temperature.

(iii) CCR Cryostat:

When handling of liquid cryogen is not desired or obtaining these is difficult, a mechanical refrigerator is used to cool down the system. Objects may be cooled by attaching them to a metallic coldplate inside a vacuum chamber which is in thermal contact with the helium vapour chamber.

1.6 Why Go Low:

Low temperature has high implications. The very wide range of temperature accessible to experiments has made temperature probably the most important among the parameters which can be varied in the laboratory in order to change the properties of the matter, to obtain a better understanding of its behaviour, and to make practical use of it.

The following few points would make clear the reasons behind going for low temperature:

- Materials are made up of atoms. These atoms are always in motion. But when we bring down the temperature then these vibrations begin to cease. The materials

become more & more ideal or “simpler”; they approach their theoretical models more closely. These models are nothing but a static picture of a system. So low temperature aids us to validate a theoretical model. At low temperature the number of excitations decreases & the vibrations of the atoms can be described in the harmonic approximation. If the potential V is a function of distance $r-r_0$ from the equilibrium position r_0 , then it can be written as,

$$V(r-r_0) \propto (r-r_0)^2$$

In this approximation there is no thermal expansion because thermal expansion results from anharmonic part of the potential. Therefore, the thermal expansion coefficient becomes smaller & smaller as we approach lower & lower temperatures.

- At high temperatures the responses of the properties are mixed. As excitations are no longer interactive at low temperature, one can specifically study a phenomenon independently. For example, the temperature dependence of the electrical resistivity of the metal just results from the interaction of the conduction electrons with the lattice. At low temperature if $\sigma_1, \sigma_2, \sigma_3, \dots, \sigma_n$ be the conductivity of the individual atoms then the total conductivity of the material is given by,

$$\sigma = \sigma_1 + \sigma_2 + \sigma_3 + \dots + \sigma_n$$

The correlation effect of electrons can be clearly seen at low temperatures.

- Electrons in a metal get scattered by lattice vibrations, defects & by other electrons. In a metal, lattice vibrations dominate the scattering at high temperature. The mean free path of the electron varies inversely as the temperature. But at very low temperatures residual impurities in a metal dominate the scattering. When we consider multiple scattering at defects, it turns out that there can be constructive & destructive

interference occurring between electron waves travelling from one point to another along different paths. This interference causes an increase in resistivity of the material as the temperature is reduced. This is called weak localisations & the effect is only seen at low temperature.

- The much talked about phenomenon “Superconductivity” was discovered in 1911 by the Dutch physicist, Heike Kammerlingh Onnes in his pursuit of exploring extremely cold refrigeration. To study this phenomenon one needs to attain low temperature.

1.7 The Present Picture:

The micro kelvin range has also been realised with the advent of nuclear demagnetisation. But the quest to reach as close as to absolute zero is still on.

Studies of materials at the lowest attainable temperatures provide key insights into the underlying physics whose applications often lead to new technological breakthroughs. The study of physicochemical structure of solid state and connection to physical, chemical and spectroscopic properties at low temperatures form a major part of cryogenic research these days. And other hottest themes are high T_c superconductors, exotic states of matter (e.g. Bose-Einstein condensate) & magnetism and electronic properties, transport and phonon phenomena at cryogenic domain. And researchers are also developing theoretical models to study the physics of low temperature plasma at low temperatures.

1.8 Motivation:

When a cryogen is transferred to the cryostat from a storage dewar, a huge amount of loss is incurred due to the boil off during the transit. And along the transfer line a layer of moisture gets deposited which increases the heat load on the transfer tube and hampers the transfer

process. Also each time the liquid is transferred, in the first few hours of transfer liquid gets used up in cooling the system only.

Keeping these considerations in view, the idea in this work is to integrate a measurement system with a liquefying unit so that transfer process can be done away with. The integration means designing a N₂ liquefier cum a general purpose cryostat (a bath type). Conventionally the liquefier & the cryostat are discrete units. Here the attempt is to accommodate both the CCR & the sample chamber in one system. This means an additional mechanical as well as thermal load on the system. The plan is to simply try out a design & do the heat transfer calculations to see whether such arrangement is practicable or not.

2-PRINCIPLES OF CRYOSTAT DESIGN

2.1 Engineering Design, An overview:

There are two approaches in engineering design. They are:

a. Allowable Stress Design Method:

It is based on the yield stress (Elastic material) or other predetermined strain amount (for a non-elastic material). The designer ensures that the stresses developed in a structure due to service loads do not exceed the elastic limit. This limit is usually determined by ensuring that stresses remain within the limits through the use of factors of safety. That permissible stress is called allowable stress.

Every material has its own capacity to hold a certain amount of load. But it's never loaded to its full capacity, because it needs to have reserve strength for following reasons:

- i. It may experience a load greater than anticipated.
- ii. Material may be defective.
- iii. Defect in fabrication.
- iv. Other unforeseen situation (like calculation errors)

Therefore the factor of safety is applied that provides a margin for errors & uncertainty. This is the earliest & most traditional design method, and also least involved computationally.

b. Limit State Design Method:

It's based on the ultimate strength of the material. Instead of reducing the allowable stress, the ultimate strength of the material is utilised applying multipliers to load. It works because mostly we are knowledgeable about the material capacity more accurately than the anticipated loads.

In this the optimum use of material strength is possible. It is more rational than the previous approach. It can lead to material saving by a considerable amount. But it is more demanding; one needs to do laborious calculations. It's mostly suitable to large scale projects.

2.2 Shape:

Mostly cryostats are cylindrical in shape. It's because it is the most economical configuration. Other possible shapes are spherical, cylindrical & conical. Spherical shape is the most effective configuration as far as the heat leak is concerned because it has the least surface area. But when it comes to fabrication, cylindrical vessels are the best from ease of fabrication point of view. To construct a cylindrical vessel, a sheet of SS is rolled and closed with either flat or curved sheets at the top and bottom ends. The common end heads are flat dish, elliptical or hemispherical.

2.3 Construction & Mechanical Reliability:

The properties of materials change very significantly when they are exposed to low temperature. Hence for construction of cryogenic apparatus, the materials are to be selected carefully. The most commonly used materials in construction are stainless steel, copper, aluminium, brass. All these materials don't become brittle at low temperature. Stainless steel has a large strength to weight ratio & one can use thin walled tubes for construction. This

reduces the weight of the cryostat. The superior heat & corrosion resistance, low thermal conductivity, high formability, aesthetic appearance & low maintenance have made the SS one of the mostly used material in cryogenics. Also it is non-magnetic. And at the places where high conductivity is required e.g. sample holder, the copper is used.

Since in cryogenic construction, we may have to use dissimilar materials. One has to take ample care about differential contraction which may lead opening of joints leading to leaks.

The thermal expansion per unit length is, $\Delta L/L_0 = \int_{T_1}^{T_2} \alpha dT$...eqⁿ (i)

Where, L_0 is the length at temperature T_1 ,

ΔL is the change in length after reaching temperature T_2

So in making a joint the material with the higher contraction should be outside the material with the lesser contraction. So on cooling the outer tube will contract more & squeeze the inner tube thus tightening the joint. Also for sealing & mounting at low temperature, some specific materials are used.

2.4 Heat Transfer:

In general heat is transferred by conduction, convection & radiation. For an efficient system we need to minimize heat losses from these sources.

Effective heat transfer takes place by:

- ❖ Conduction through the residual low pressure gas.
- ❖ Conduction through the solids that interconnect the various parts of the cryostat.
- ❖ By radiation

Conduction through gas:

In cryogenic situation, the gas pressure (P) is nearly always reduced to a point where the mean free path is comparable with the dimension of the system. This is called Knudsen conditions. Then the average molecule travels from hot wall to cold wall without collision with another gas molecule & the thermal conductivity (K) becomes a function of the number of the molecules (n) & their velocity, i.e. at low pressure $K \propto n \propto P$.

For approximately parallel surfaces at temperatures T_1 & T_2 , ($T_1 < T_2$), the heat transferred from hot walled to cold wall by conduction through the gas is given by,

$$\dot{Q} = a_{avg} \left(\frac{\gamma+1}{\gamma-1} \right) \sqrt{\frac{R}{8\pi m}} P \frac{T_2 - T_1}{\sqrt{T}} \quad \dots \text{eq}^n \text{ (ii)}$$

Where, M = molecular weight

$$\gamma = \frac{C_p}{C_v}$$

R= gas constant

T= temperature of the pressure gauge

a_{avg} is an average over the individual accommodation coefficients, a_1 & a_2 , for the gas molecules at the two surfaces.

Accommodation coefficient is a physical quantity characterizing the behaviour of gas or vapour particles in their collisions with a solid or liquid body surface. If two surfaces have roughly equal area A, then

$$a_{avg} = \frac{a_1 a_2}{a_1 + a_2 - a_1 a_2} \quad \dots \text{eq}^n \text{ (iii)}$$

Again if these individual coefficients are roughly equal, then $a_{avg} = \frac{a_1}{2}$ as a_1 tends to zero and $a_{avg} = a_1$ as a_1 tends to 1.

Accommodation coefficients are normally measured for the case of a wire at temperature T_2 which is slightly greater than a surrounding gas at temperature T_1 . In this case molecules colliding with the wire have an average energy corresponding to T_1 . The molecules leave the wire with an energy corresponding to a temperature T'_2 intermediate between T_1 & T_2 ; then

$$T'_2 - T_1 = a(T_2 - T_1) \text{ defines the accommodation coefficient 'a'.$$

In eqⁿ (ii), it is impossible to identify 'T' with either T_1 or T_2 . According to Corruccini, 'P' is observed at vacuum gauge usually at room temperature. Under low pressure conditions the free path $l \gg d$, the diameter of the tube connecting the gauge to the cryostat chamber. If the system is in equilibrium, & a value of P obtained from the gauge is inserted in the eqⁿ (ii), the appropriate value for T is the temperature of the pressure gauge.

So, assuming $T = 295K$, eqⁿ (i) gets simplified to

$$Q \approx \text{constant} \times a \times P(T_2 - T_1) \quad \dots \text{eq}^n \text{ (iv)}$$

$$\text{Where the constant is approximately } 1.0 \times \frac{\gamma+1}{\gamma-1} \sqrt{\frac{R}{8\pi MT}}$$

The value for this constant has been calculated to be 1.2 for air & 2.1 for Helium with \dot{Q} in Wm^{-2} & P in Pa.

Conduction through solids:

The heat flow through a solid of cross section A under a temperature gradient dT/dx is given by,

$$\dot{Q} = K(T)A \frac{dT}{dx} \quad \dots \text{eq}^n \text{ (v)}$$

If the ends of a solid bar of uniform cross section & of unit length 'l' are at temperatures T_1 & T_2 , then

$$\dot{Q} = \frac{A}{l} \int_{T_1}^{T_2} K(T) dT \quad \dots \text{eq}^n \text{ (vi)}$$

The information often needed for practical cryogenic calculations is the effective heat conductance of a construction material with particular end temperatures. Common pairs of end temperatures may be 300K & 77K, 300K & 4.2K, 77 & 4.2K, 4.2K & 1K.

Mean heat conductivity is given by:

$$K_{avg} = \frac{1}{T_1 - T_2} \int_{T_1}^{T_2} K(T) dT \quad \dots \text{eqn (vii)}$$

Heat transfer by radiation:

For two parallel surfaces, each of area A & emissivity ϵ_1, ϵ_2 at respective temperatures T_1 & T_2 , the rate of heat transfer by radiation is,

$$\dot{Q} = \sigma A (T_1^4 - T_2^4) \frac{\epsilon_1 \epsilon_2}{\epsilon_1 + \epsilon_2 - \epsilon_1 \epsilon_2} \quad \dots \text{eq}^n \text{ (viii)}$$

where σ = Stefan's constant = $5.67 \times 10^{-8} \text{ Wm}^{-2}\text{K}^{-4}$

The heat is propagated as electromagnetic waves & the energy will be absorbed by the second surface on which the radiation is incident. The absorption coefficient of a surface is equal to its emissivity. So the larger value of ϵ means the larger quantity of heat is absorbed. The emissivity of a surface can be reduced by polishing the surface or even plating the surface with materials having low emissivity. In very low temperature work, the parts are coated with gold to reduce heat load due to radiation.

2.5 Storage of Liquefied Gas:

The liquefied gas is at a temperature much below the ambient temperature. So heat will leak from the surrounding to the cryogen. This makes the cryogen to boil & evaporate. To keep the evaporation rate at minimum, one has to reduce the heat leak from all those processes mentioned above.

A double walled container serves the purpose well. The inner chamber is used for storing liquid cryogen. And the space between the two walls is evacuated to a pressure of 10^{-6} mbars & sealed. So in low temperature applications, convection is not significant because of the partial evacuation of the gas from the interior of the cryostat.

To reduce heat leak further intermediate shields between the warm & cold surfaces can be introduced. The number of layers to be wound & the layer density are determined by the competition between radiation heat leak & heat leak through solid contact conduction. As the number of layer increases, the radiation heat leak decreases. To make the windings compact one would like to have as many layers per centimetre thickness as possible. As the layer density increases, heat transfer through solid contact conduction increases. This ultimately limits the layer density. And also radiation baffles made up of copper just below the lid of the cryostat is used to minimise the radiation inflow which are supported by solid rods of SS.

Another contribution to the heat leaking into the cryogen comes through heat conduction from the top of dewar through the neck. To reduce this heat, one chooses a material that has a low thermal conductivity & high mechanical strength. The Stainless Steel makes it possible. The high mechanical strength of this material allows us to choose a small wall thickness (about 0.2 to 0.5 mm) for inner container.

3 - THE DESIGN

3.1 Design Considerations:

- i. **Purpose:** Mainly R-T measurement. In the beginning we can't be very ambitious. If the design seems to work out, then the idea may be extended to the measurement of other properties as well.
- ii. **Operating Range:** 300-77K.
- iii. **Type:** A CCR cooled bath cryostat. The gas will be liquefied with the aid of the CCR which will be stored in the vacuum dewar. A measurement system will be immersed in this container. It will be a sample chamber that will be welded & the elongated tube will run right into the liquid cryogen.
- iv. **Temperature Profile:** Can be both fixed and variable.

In a bath type generally the temperature remains fixed to within a few millikelvins because the liquid boils off at a fixed temperature at atmospheric pressure. The temperature can be lowered below the atmospheric boiling temperature by evacuating the space above the cryogen. This reduces the boiling point of the entire bath of the liquid. And at elevated temperature, the dewar vent is sealed off. It causes the evaporating liquid to pressurise the vessel. This increases the boiling point of the cryogen. But we need to be cautious about it, because overpressure can blow up the dewar. Generally it is advised not to go above 2 atm.

Also in pressurizing the dewar, "Temperature Stratification" occurs. The liquid at the top is at higher temperature whereas a colder liquid sits at the bottom. To keep the temperature uniform, a resistive heater is placed at the bottom of the vessel.

- v. Access to the experiment: Electrical leads of heater, 4 probe & temperature sensor.
- Well here we would use a dipstick which will be inserted into the sample chamber for a measurement. The dipstick rod will have a specific part for containing the sample. And with a space where the heater wires can be wound.

3.2 Design Description:

The fig. 3.1 above shows the liquefying unit of CRYOMECH LNP40. This Liquid Nitrogen Plant is available in the Cryogenic Centre of NIT Rourkela. The CRYOMECH Inc. offers this as a standard configuration. It has production rate of about 40 litres/day. This has been used as a reference here.

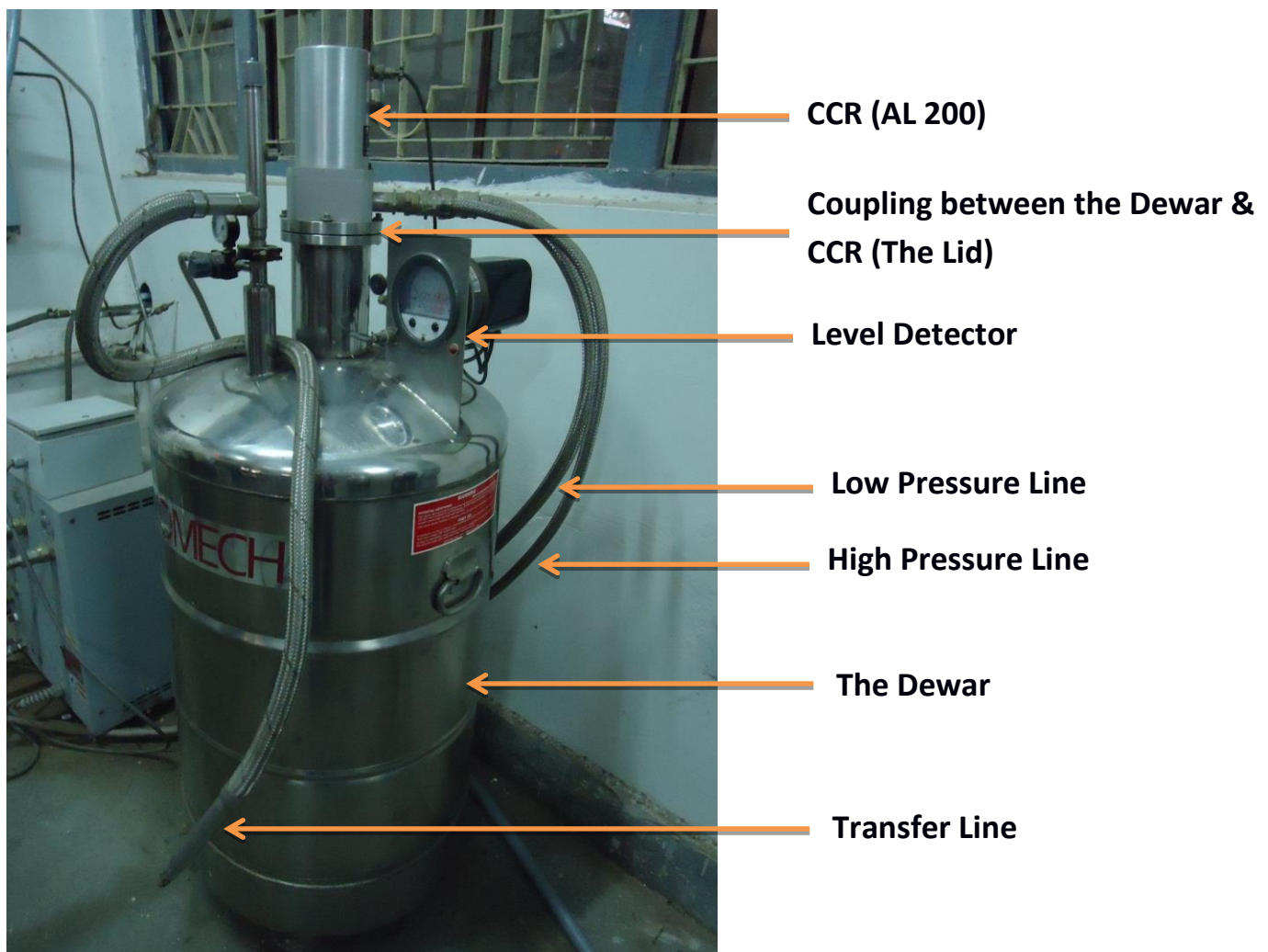


Fig 3.1: Existing closed cycle based liquid nitrogen plant

We plan to add in a measurement system in the lid. With the new added component & some few modifications the system would look somewhat like as follows (Fig. 3.2):

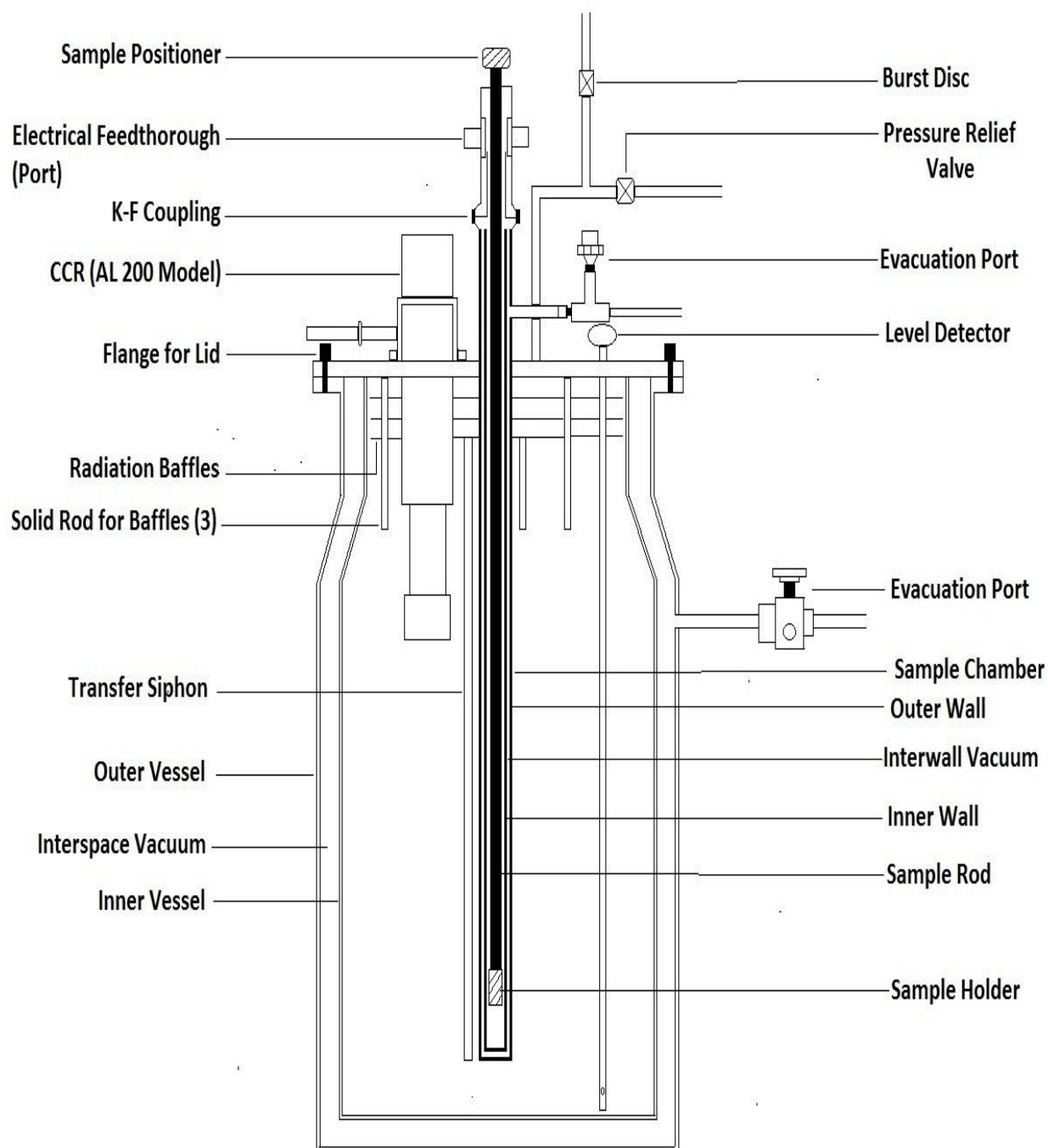


Fig 3.2: Schematic of the N₂ Liquefier with Measurement Cryostat Based on CCR

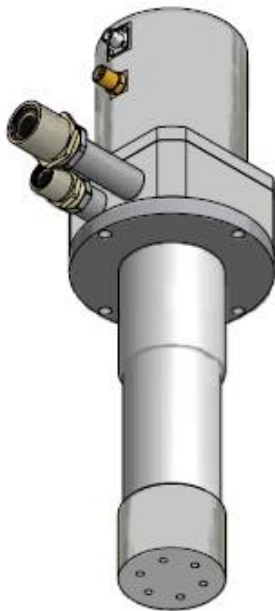
The above diagram depicts the design to be followed. The different parts are as given below:

➤ **The Dewar:**

It's the double walled cylindrical container to hold the liquid cryogen. It is made of stainless steel.

➤ **CCR:**

A cryorefrigerator (here GM based) is used for liquefying Nitrogen. It's the AL200 Cold Head from CRYOMECH.



It is attached to the top of the Dewar with 4 bolts. An O-ring, located between the dewar flange & the CCR seals the Nitrogen gas inside the dewar. The nitrogen condenses on the cold end of the cryorefrigerator & drips to the bottom of the dewar.

Fig 3.3: CCR (AL 200)

➤ **Pressure Relief Valve:**

This is required for venting out the gas when the pressure inside the dewar reaches a certain value. This is very important in protecting the dewar from overpressure. Generally it is advised to have 2 pressure relief valves in a system.

➤ **Burst Disc:**

If somehow the relief valves fail, then it will burst & release the cold gas to the atmosphere. It's an additional safety feature.

➤ **Level detector:**

It monitors the amount of liquid & regulates the amount of liquid in the dewar. It may also work as a preset to shut down the system when the dewar is full. Generally for nitrogen capacitive detectors are good. The American Magetics Inc. provides a wide range of capacitive level detectors to be used with digital monitors & controller.

➤ **Sample Chamber:**

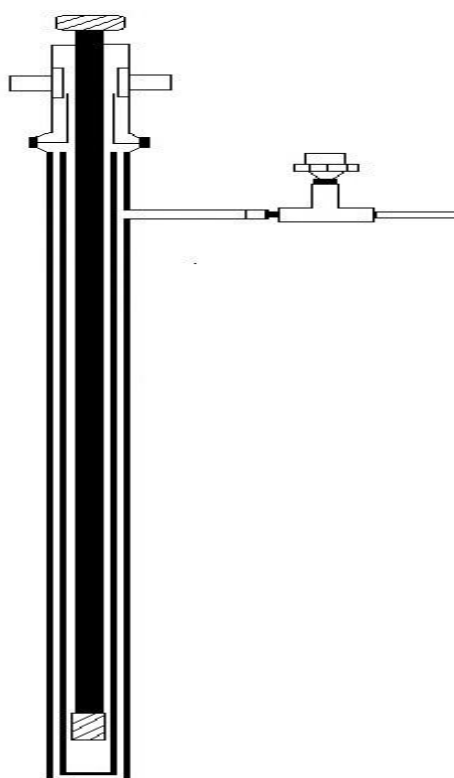


Fig 3.4: Measurement Unit

This is the measurement system which is an added component to the top of the dewar. This is also a double walled cylindrical structure. The inner walls run up to the K-F coupling. It has ports for electrical feed through. A SS rod with a sample holder (copper) is inserted inside this whenever a measurement needs to be done. The positioner helps to adjust the height. It's an arrangement with a knob which when rotated in a certain direction tends to give us the desired position. It has

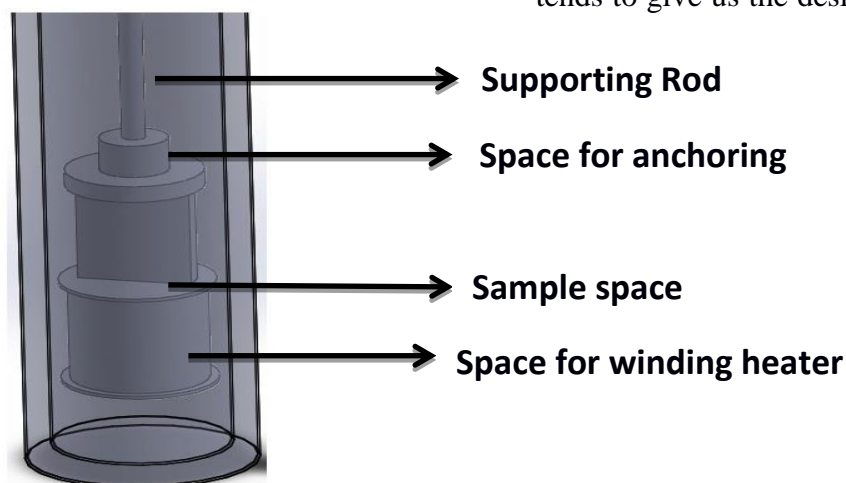


Fig 3.5: Sample Holder

As we intend to do R-T measurement, we normally use a heater whose leads are wound around the sample holder. If the vacuum isn't there then a considerable amount of heat from the sample chamber will flow towards the cryogen. This will result in boiling off of the liquid. It's a compromise because the presence of vacuum also delays the cooling process of the sample. But as far as the storing of the cryogen is concerned, it's a fair deal. Also we can have a needle valve in the chamber for cooling by exchange gas if we need a faster cooling of the sample.

Electrically leads need to be heat sunk to the sample holder before attaching to the sample & thermometer in order to intercept heat conduction along the lead from the room temperature. This ensures that the sample, sample holder & thermometer are all at the same temperature.

➤ **Evacuation Port:**

There is a side evacuation port for creating the inter space vacuum. The sample chamber also contains an evacuation port.

3.3 Fixing the Dimensions:

The length & outer diameter values were taken from CRYOMECH LNP 40 specifications. It gives,

$$D_0 = 610 \text{ mm}, L = 1520 \text{ mm}$$

Taking these as starting lead, we get the following dimensions. The thickness of the outer, inner vessel & the lid is very crucial as the stability of the whole system depends on it. These were calculated as follows:

Thickness Calculations :

The calculations were based on ASME (American Society of Mechanical Engineers) Boiler & Pressure Vessel Code Section- VIII.

ASME is a non-profit professional society that endorses art, science & practice of

multidisciplinary engineering & allied sciences around the globe by imparting training, project development codes & standards and through other various resourceful approaches.

BPVC is a standard that provides rules for design, fabrication & inspection of boilers & pressure vessels.

- A cylinder is defined as long, when its length to diameter ratio meets the following condition,

$$L/D_0 > 1.140 * (1 - \gamma^2)^{0.25} * (D_0/t)^{0.5} \quad \dots \text{eq}^n (\text{ix})$$

Where,

L = Length of the cylinder

D_0 = Outer diameter

t = Thickness

γ = Poisson's Ratio

Otherwise it is a short cylinder.

In this section we'd try to estimate the thickness of the three most important components of the cryostat namely vessel's wall, sample chamber & the lid.

[I] Vessel's thickness:

(i) Outer Vessel Thickness

The collapsing pressure for a short cylinder is given by ,

$$P_c = \frac{2.42 * Y * (t/D_0)^{2.5}}{(1 - \gamma^2)^{0.75} * [L/D_0 - 0.45 * (t/D_0)^{0.5}]} \quad \dots \text{eq}^n (\text{X})$$

Where,

Y = Young's Modulus of the material used

t = thickness of the vessel

D_0 = Outer Diameter of the vessel

γ = Poisson's ratio

- The length & outer diameter values were taken from CRYOMECH LNP 40 specifications. It gives,

$$D_0 = 610mm, L = 1520mm$$

With these dimensions, we can readily show why we're taking the vessel to be a short one. Here it how it goes:

We've $L/D_0 = 2.5$ & the right hand side of the eqⁿ(ix) gives us

$$1.140 \times (1 - 0.26^2)^{0.25} \times \left(D_0/t \right)^{0.5} = 12.3673 \quad (\text{taking } t=5mm)$$

So, definitely $L/D_0 < 12.3673$. Here we did guesswork for the thickness just to reach to a value for the left hand side. As RHS value is quite high compared to the LHS, so even a higher value of thickness would lead to same reasoning. And a lower value of thickness would result in a still greater value for the same. So in both instance we'll head towards the same result.

(N.B.: One may feel that it's somehow inaccurate to conclude in such a way, but in true experimental spirit sometimes we've to pursue "seat-of-the-pants" style. In fact you would come across this a lot of time in my work.)

- The value of collapsing pressure is determined from,

$$P_c = 4 * P_a$$

Where, P_a is allowable external pressure (equal to atmospheric pressure).

We know, $P_a = 100 \text{ KPa} = 0.1 \text{ N/mm}^2$

Therefore we have, $P_c = 4 * 0.1 = 0.4 \text{ N/mm}^2$

- For Stainless Steel,

$$\gamma_{ss} = 0.26, \quad Y_{ss} = 19.3 \times 10^4 \text{ N/mm}^2$$

Now from eqⁿ (x), we have :

$$(t/D_0)^{2.5} = \frac{P_c * (1 - \gamma^2)^{0.75} * [L/D_0 - 0.45 * (t/D_0)^{0.5}]}{2.42 * Y}$$

Putting all the values we obtain,

$$t^{2.5} (1/610)^{2.5} = \frac{0.4 * (1 - 0.26^2)^{0.75} * [2.5 - 0.45 * t/D_0]^{0.5}}{2.42 * 19.3 \times 10^4}$$

$$\Rightarrow t^{2.5} * (1.0881 \times 10^{-7}) = \frac{0.4 * 0.9488 [2.5 - 0.45 * 0.0405 * t^{0.5}]}{46.706 \times 10^4}$$

$$= \frac{0.9488 - 0.0069 * t^{0.5}}{46.706 \times 10^4}$$

$$\Rightarrow \boxed{t^{2.5} * 0.0508 + 0.0069 * t^{0.5} - 0.9488 = 0} \quad \leftarrow f(x) \quad \dots \text{eq}^n \text{ (xi)}$$

Now, we shall calculate the root of the above equation by using Newton-Raphson Method.

According to this method the nth root of the equation is given by,

$$x_{n+1} = x_n - \frac{f(x_n)}{f'(x_n)} \quad \dots \text{eq}^n \text{ (xii)}$$

Here $f(x)$ is given by eqⁿ (xi).

$$\text{So, } f'(x) = 0.0625 * t^{1.5} + 0.0079 * t^{-0.5}$$

➤ To find out the limits we proceed as follows:

Putting different arbitrary values for 't' in $f(x)$, we get

$$f(3) = -0.145, f(3.5) = 0.2283$$

So this means that the root lies in between 3 & 3.5.

1st Iteration:

$$x_0 = 3$$

$$x_1 = 3 - \frac{(-0.145)}{0.6619} = 3.2191$$

2nd Iteration:

$$x_1 = 3.2191$$

$$x_2 = 3.2191 - \frac{0.0081}{0.7354} = 3.2081$$

After these two iterations we can well take the vessel's thickness to be 3.3 mm which is an upper bound on the value.

Thus thickness of outer vessel,

$t_o = 3.3 \text{ mm}$

(ii) Inner Vessel Thickness

We can't use the same formula which we used earlier because of the fact that the pressure here acts inside out whereas for the outer vessel it acts outside in. So the outside vessel is likely to collapse while the inner one is prone to bursting.

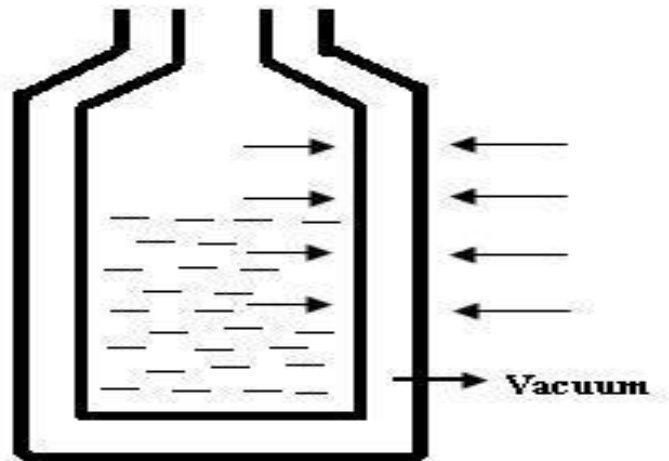


Fig 3.6: Pressure Profile in a double walled cryogenic container

The minimum thickness of the inner cylinder is decided by,

$$t = \frac{p * D_0}{2 * S_a * e_w + 0.8 * p} \quad \dots \text{eq}^n \text{ (xiii)}$$

Where, p = Design pressure

S_a = Allowable stress

e_w = Weld efficiency

- Here the design pressure which is the absolute pressure for the vacuum jacketed vessel would be the difference between the pressure inside the inner chamber & the vacuum following it.

Degree of vacuum = 10^{-6} mbar

Pressure inside the inner chamber = 1 bar

Hence the Pressure difference = 1 bar – 10^{-6} mbar

$$= (1 - 10^{-9}) * 10^5 \text{ N/mm}^2$$

$$= 0.1 \text{ N/mm}^2$$

$$\Rightarrow p = 0.1 \text{ N/mm}^2$$

- Allowable stress for stainless steel (304 welded tube) ,

$$S_a = 16,000 \text{ psi}$$

$$= 16,000 * 6894.757 \text{ N/m}^2$$

$$= 110316112 * 10^{-6} \text{ N/mm}^2$$

- Welding efficiency gives the ratio of strength of a welded or brazed joint to the strength of the parent material. It depends upon the type of the joint & radiography.

-when not radiographed, $e_w \approx 60 \%$

Spot radiographed, $e_w \approx 85 \%$

Fully radiographed, $e_w \approx 100 \%$

Here we shall take it to be 60%.

Now with all these values placed in eqⁿ (xiii), we acquire:

$$t = \frac{0.1 * 610}{2 * 110316112 * 10^{-6} * 0.6 + 0.8 * 0.1}$$

$$= \frac{61}{10^{-6} * 132379334.4 + 0.08} = \frac{61}{132.3793} = 0.46 \text{ mm}$$

As we did in the case of the outer vessel, on the safe side we can take a higher value for the thickness here.

Hence thickness of inner vessel,

$t_i = 0.5 \text{ mm}$

➔ We can also follow another approach for the thickness calculation using eqⁿ (x) similar to what we did for the outer vessel. It goes as follows:

We know, $L = 1520$ mm, $D_0 = 610$ mm, $t_0 = 3.3$ mm.

Let the interspace vacuum be there for 30 mm i.e. the gap between the inner chamber & the outer one is 3 cm.

If D_i is the diameter of the inner chamber, then we have:

$$\begin{aligned} D_i &= D_0 - (2 \times 20 + 2 \times 3.3) \\ &= 610 - 66.6 \\ &= 563.4 \text{ mm} \end{aligned}$$

Let the length of the inner chamber be 1000 mm. And here $P_c = 0.1$ N/ mm²

With all these values the eqⁿ (ii) gives us,

$$\begin{aligned} 0.1 &= \frac{2.42 * 19.3 * 10^4 * (t/563.4)^{2.5}}{(1 - 0.26^2)^{0.75} * [\frac{1000}{563.4} - 0.45 * (t/563.4)^{0.5}]} \\ &= \frac{6.1991 * 10^{-6} * 10^4 * t^{2.5}}{0.9488 * [1.8403 - 0.019 * t^{0.5}]} \\ &= \frac{0.062 * t^{2.5}}{1.6840 - 0.018 * t^{0.5}} \end{aligned}$$

$$\Rightarrow \boxed{0.0018 * t^{0.5} + 0.062 * t^{2.5} - 0.1684 = 0} \quad \leftarrow f(x) \quad \dots \text{eq}^n(\text{xiv})$$

$$\Rightarrow f'(x) = 0.0009 * t^{-0.5} + 0.155 * t^{1.5}$$

$$\text{Now, } f(1.5) = 0.0046, \quad f(1) = -0.1046$$

\Rightarrow The root lies between 1 & 1.5.

1st Iteration:

$$x_0 = 1.5$$

$$x_1 = 1.5 - \frac{0.0046}{0.2855} = 1.48$$

2nd Iteration:

$$x_1 = 1.48$$

$$x_2 = 1.48 - \frac{(-0.0009)}{0.2798} = 1.483$$

Again taking the upper limit on the value we can have,

$t_i = 1.5 \text{ mm}$

- There is a noticeable amount of difference between the values obtained from these two approaches. Of, course in the second approach the thickness depends on the value of the length of the chamber. So in all this can be viewed as a crude way of calculation which still delivers an appreciable value.

For the design purpose we'll stick to the thickness calculated by the previous approach because understandably we'd like to have thickness as small as possible.

[ii] Sample Chamber's Thickness

The sample chamber will also be two chambered. The dimension of the sample chamber depends on the kind of measurement we want to make & the size of the samples.

Here we take,

$$D_0 = 50 \text{ mm}, L = 1420 \text{ mm}$$

(i) Outer chamber

With the above dimensions the sample chamber comes out to be a long cylinder. We've to keep in mind that here the material that chamber is made up of will be subjected to the liquid Nitrogen temperature, as it'll remain immersed in the cryogen. So we'll use a different value for the Young's Modulus of the stainless steel.

At 77 K, $Y_{ss} = 20.8 \times 10^4 \text{ N/mm}^2$

For a long cylinder the collapsing pressure is given by,

$$P_c = \frac{2 \times Y \times (t/D_0)^3}{1 - \nu^3} \quad \dots \text{eq}^n(\text{xv})$$

Putting the respective values we'll obtain,

$$t^3 \times 8 \times 10^{-6} = \frac{0.393}{41.6 \times 10^4}$$

$$\Rightarrow t = 0.49 \text{ mm}$$

Again adhering to the higher value we take,

$$t_n = 0.5 \text{ mm}$$

(ii) Inner chamber

Here we stick to the same old formula given by eqⁿ (xiii).

Upon calculation that leads us to a value, $t = 0.03 \text{ mm}$. This seems extremely less. So we'd take a value equalling the thickness of the outer chamber (It's just an educative guess!).

Hence we have, $t_i = 0.5 \text{ mm}$

[iv] Sample Holder's Dimension:

These are fixed according the requirement of a measurement. There are no hard & fast rules. The size of the sample is a deciding factor. Fig. 3.7 shows the dimensions of different parts.

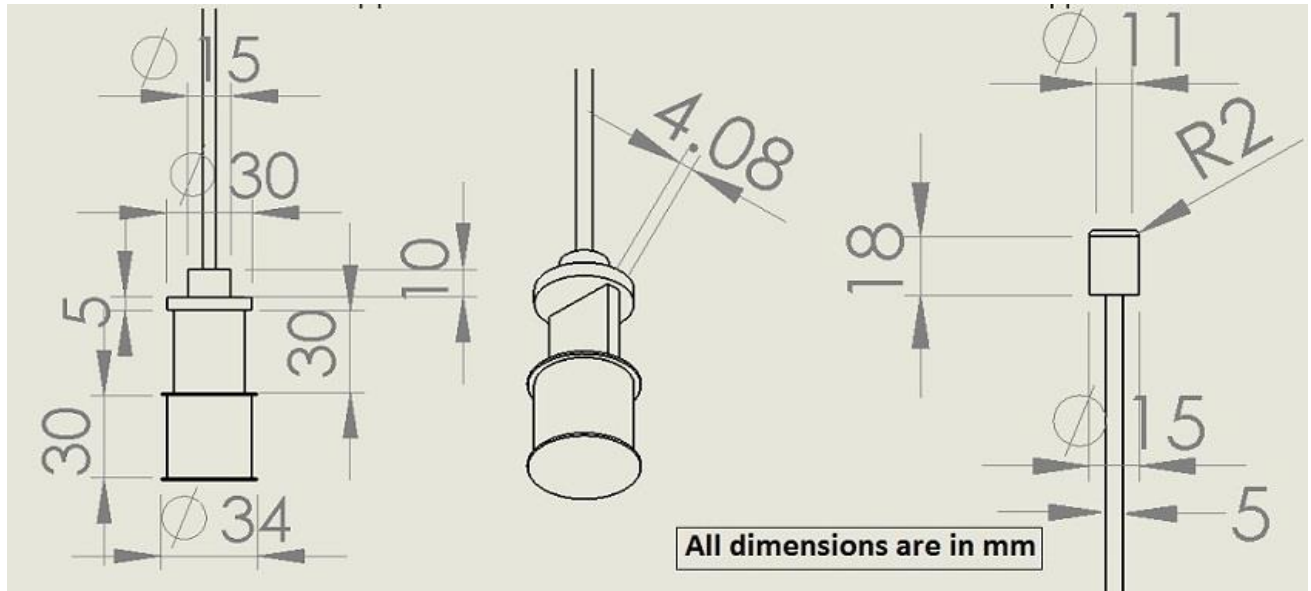


Fig 3.7: Dimension of Sample Holder

[iii] Lid's Thickness

The lid is neither centrally loaded nor uniformly loaded. That complicates the business, but anyway we'll proceed in the following way:

For a flat circular plate loaded centrally, the thickness is given by,

$$t = 1.65 \left(\frac{F}{\sigma_t} \log_e \frac{d}{d_0} \right)^{0.5} \quad \dots \text{eq}^n \text{ (xvi)}$$

Where, F = Load

d = Diameter of the whole plate

d_0 = Diameter of the area loaded centrally

σ_t = Allowable design stress

Let the rough value of total load be, $F = 20 \text{ kg}$ (Since CCR's weight = 15.4 kg)

Expressing it in terms of force we get, $F = 20 \times 9.80665 = 196.133 \text{ N}$

A top view of the lid is given in the fig. 3.8.

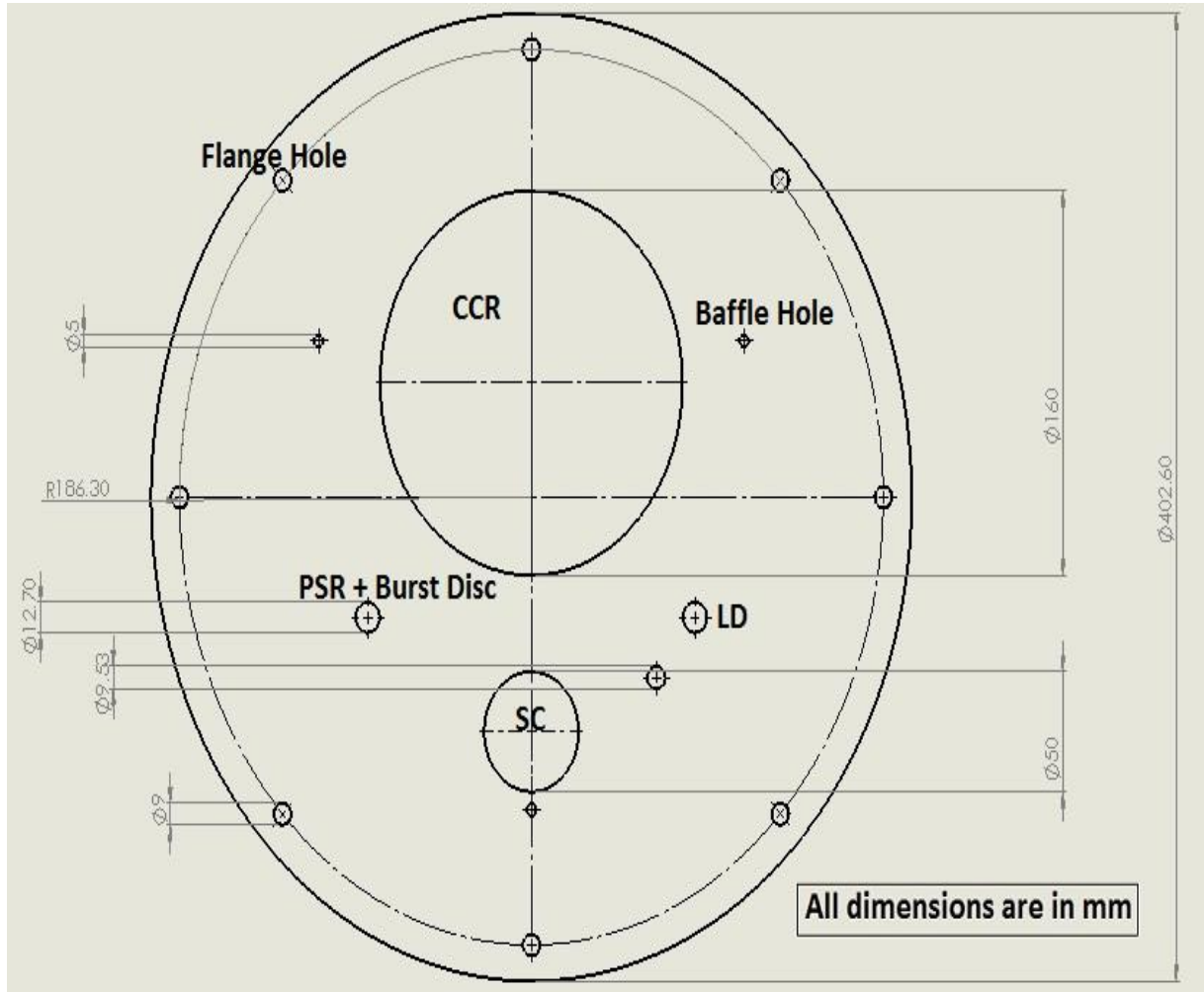


Fig 3.8: Lid View

If d_{CCR} , d_{SC} , d_{TS} , d_{LD} , d_{PR} , d_{RB} be the diameter of the area occupied by the CCR, sample chamber, transfer siphon, level detector, pressure relief valve & rod carrying baffles respectively then the diameter of area loaded will satisfy the following relation,

$$\pi \frac{d_0^2}{4} = \frac{\pi}{4} (d_{CCR}^2 + d_{SC}^2 + d_{TS}^2 + d_{LD}^2 + d_{PR}^2 + 3 \times d_{RB}^2) \quad \dots \text{Eqn (xvii)}$$

$$= (16^2 + 5^2 + 2 \times 1.27^2 + 0.9525^2 + 3 \times 0.5^2)$$

$$\Rightarrow d_0 = 16.91 \text{ mm}$$

And we have, $d = 402.6 \text{ mm}$, $\sigma_t = 110.32 \text{ N/mm}^2$

With these values we get, $t = 1.65 \sqrt{\frac{196.133}{110.32} \log_e \frac{402.6}{16.91}} = 3.927 \text{ mm}$

This sets up the lower limit for the thickness. Now with a very makeshift guess we would pick up the thickness for our purpose, we'll take double the value of what we have obtained here.

So,

$$t_{lid} = 8 \text{ mm}$$

Fig. 3.9 shows the top view from inside. The dimension is in mm.

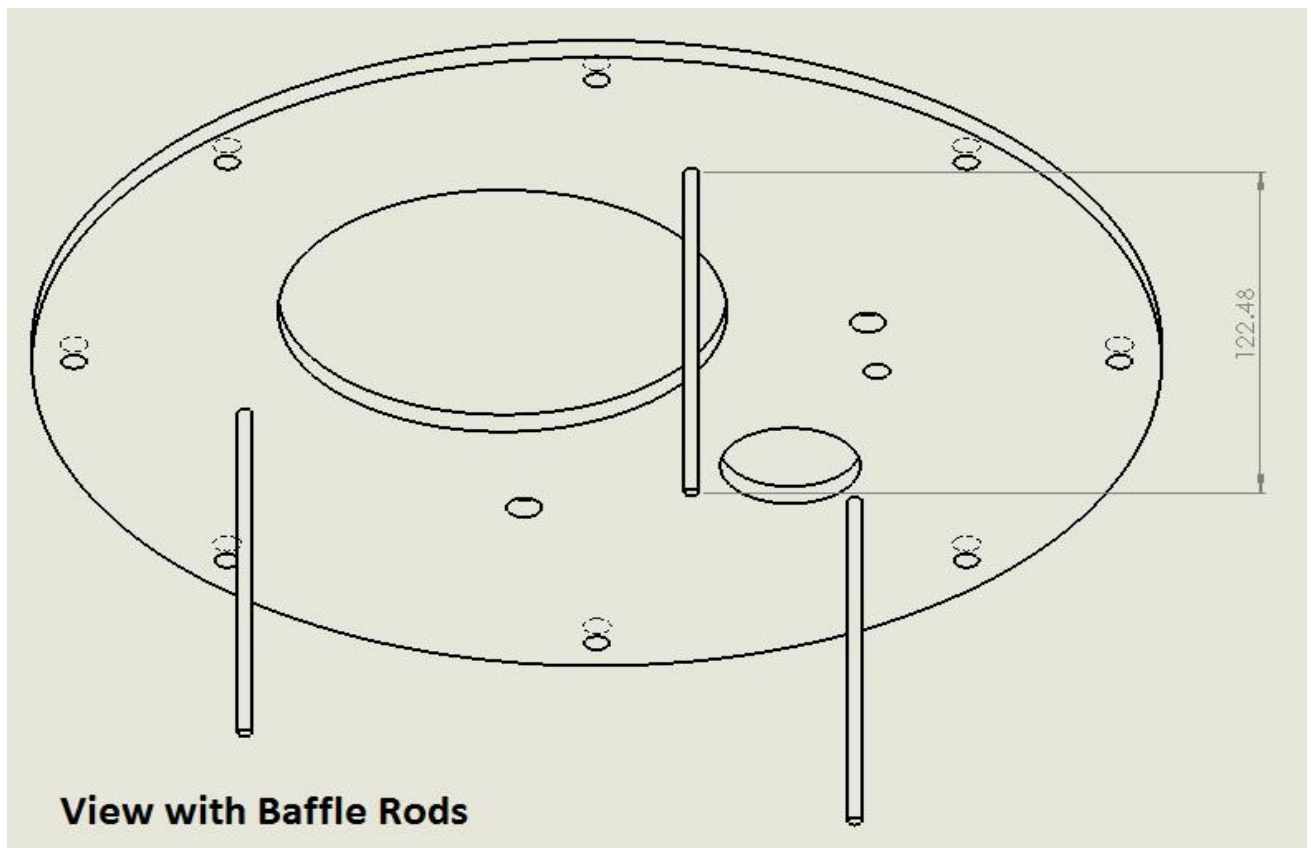


Fig 3.9: Top view with Baffle Rods

- The dimension in the fig. 3.9 is in mm.

The fig. 3.10 shows the dimensions of different parts of the system described.

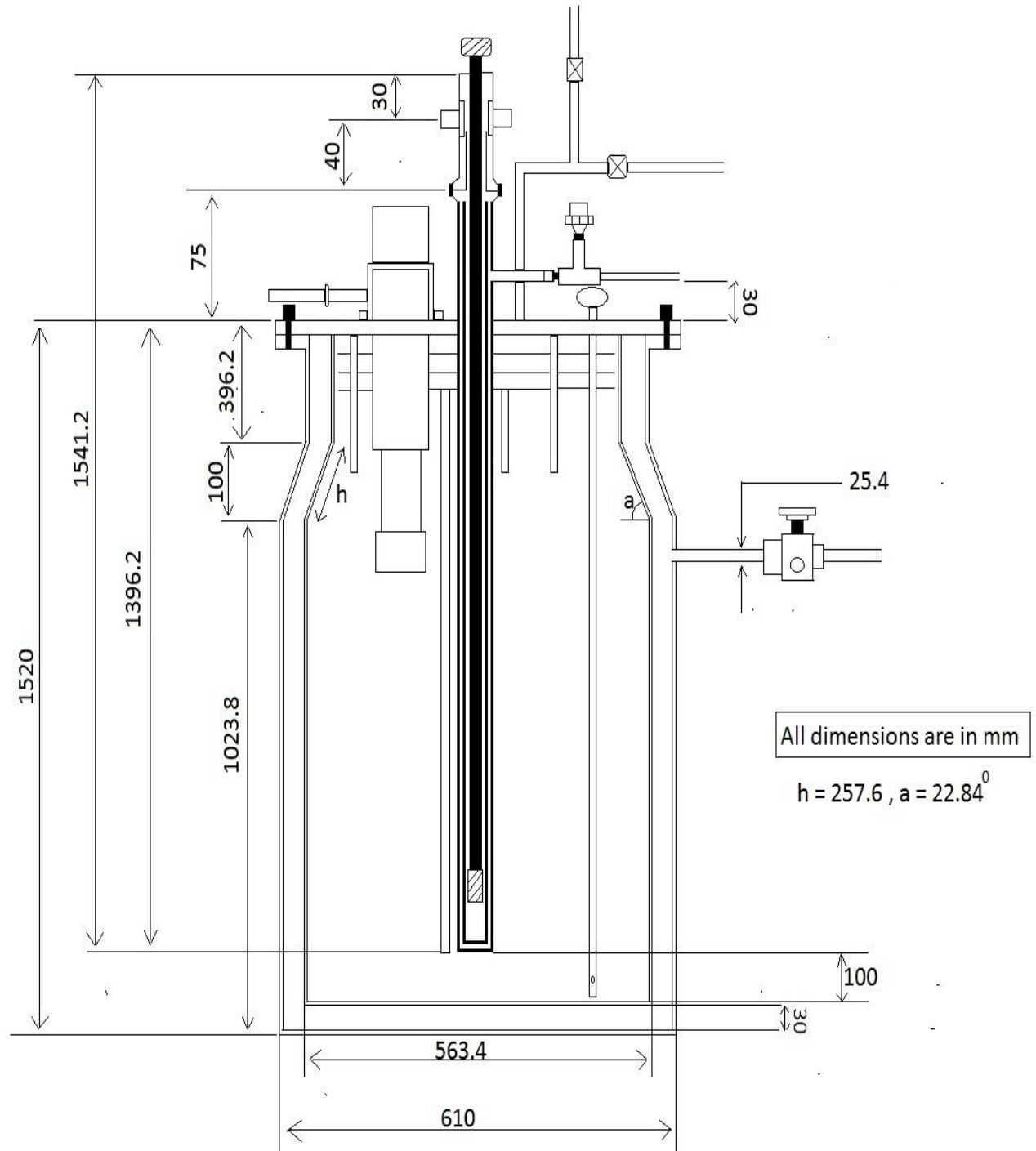


Fig. 3.10 Showing dimensions of different parts

3.4 A Solid View:

Fig.3.10 shows a simple solid view of the liquefier cum cryostat arrangement with only the two main components; CCR & the sample chamber.

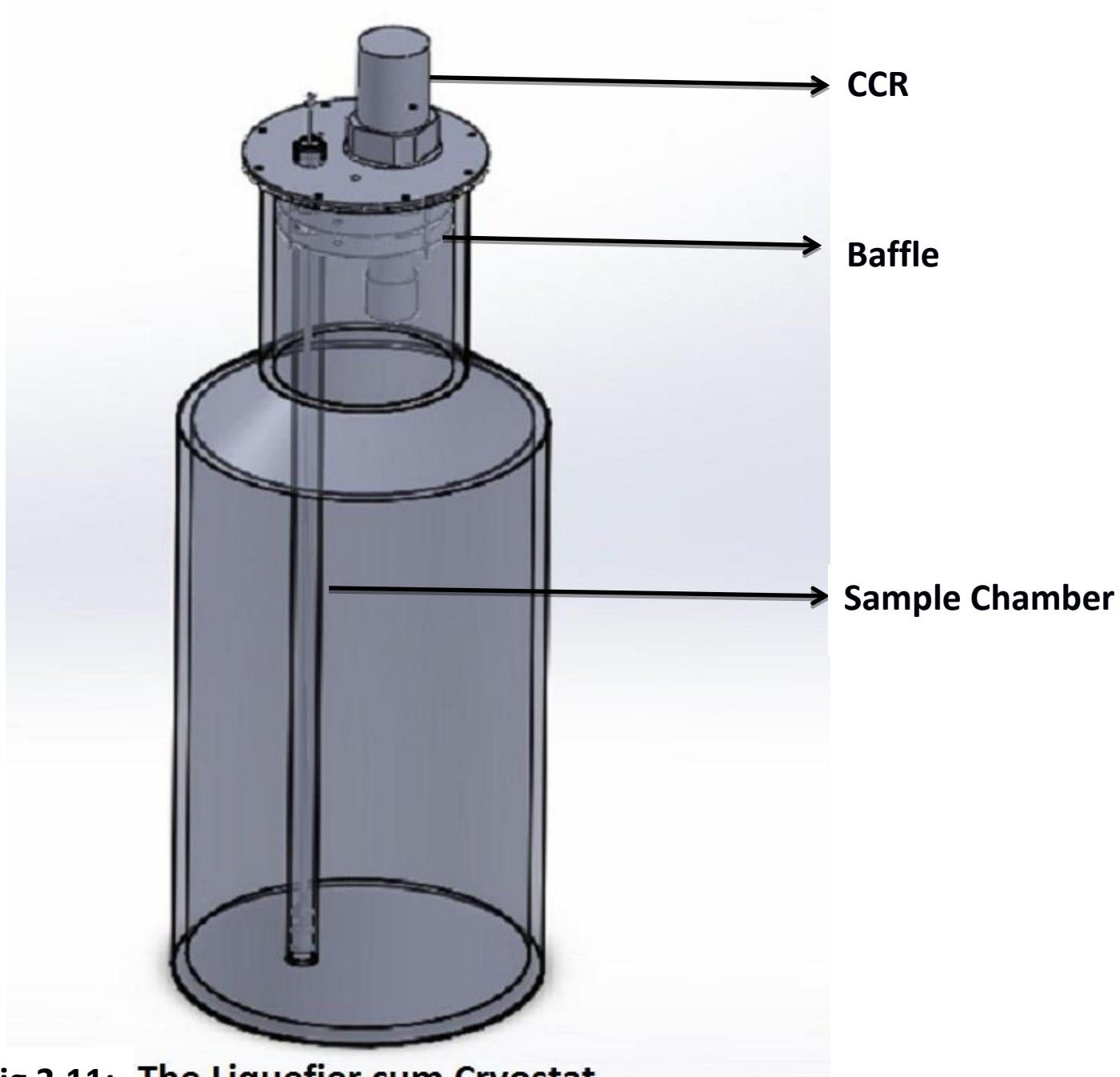


Fig 3.11: The Liquefier cum Cryostat

And fig. 3.11 gives you a close inside view of the top with radiation baffles.

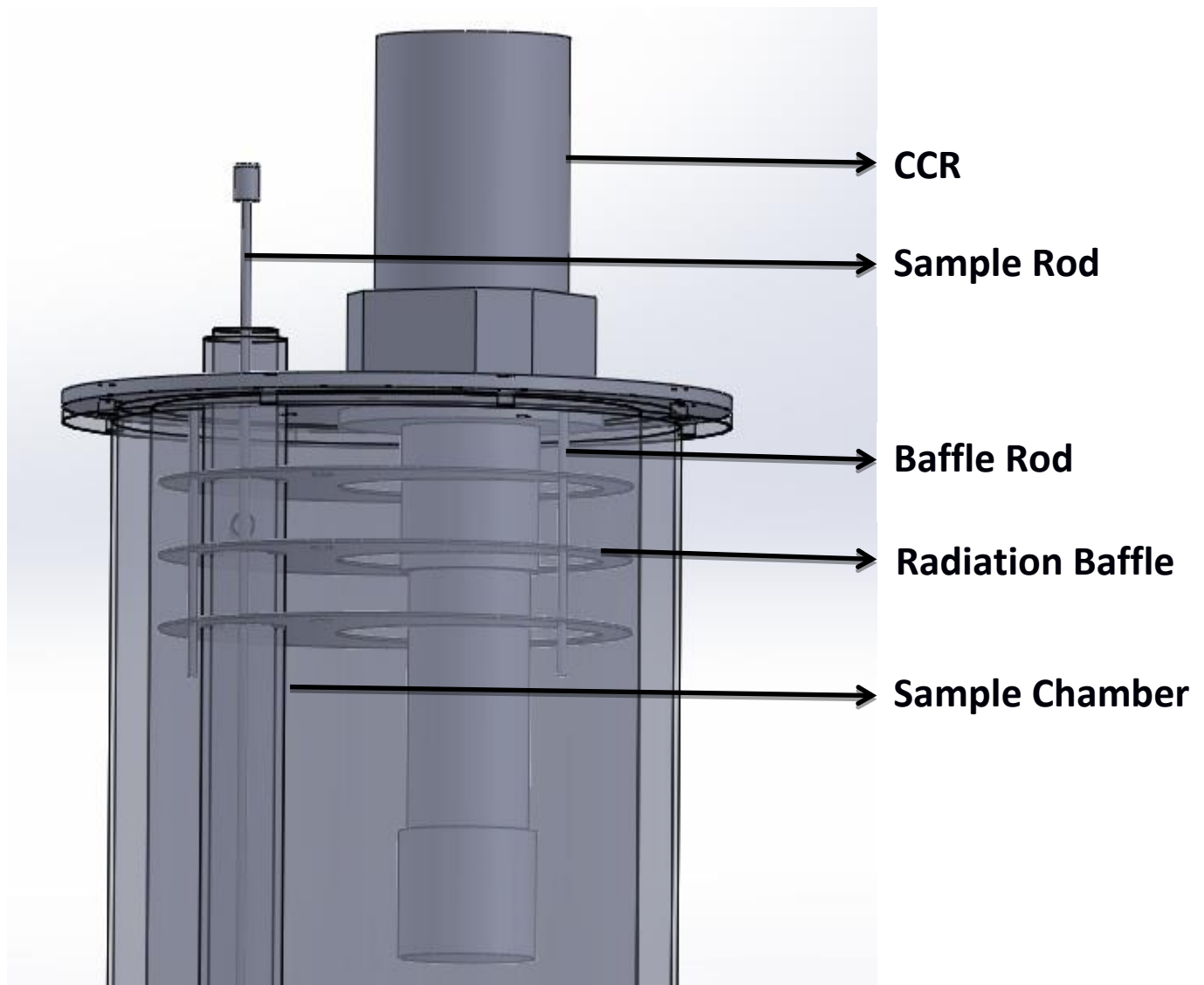


Fig 3.12: A close view of the top

4 - HEAT TRANSFER CALCULATIONS

4.1 Major sources:

The major sources of heat inflow would be those components which are directly immersed in the liquid cryogen. Those components are sample chamber, Transfer Siphon & Level Detector. Hence the sources will be as follows:

- (i) Radiant heat inflow from outer chamber to inner chamber
- (ii) Radiant heat inflow down the sample chamber (SS tube).
- (iii) Heat conducted down the sample chamber.
- (iv) Radiant heat flow down the transfer siphon (SS tube).
- (v) Heat conducted down the transfer siphon.
- (vi) Radiant inflow down the level detector (SS tube).
- (vii) Heat conducted down the level detector.
- (viii) Heat conducted down the neck of the inner wall.

4.2 The calculations:

(i) Radiant heat inflow from outer chamber to inner chamber:

Case (I) :Unpolished outer wall of inner chamber

Here $\epsilon_1 = \epsilon_2 = \epsilon_{ss} = 0.2$, $T_1 = 300K$, $T_2 = 77K$, $\sigma = 5.67 \times 10^{-8} \text{ Wm}^{-2}\text{K}^{-4}$

As the area of both the chamber are not equal, an average value will be taken.

$$A_{in} = 3.1452 \text{ m}^2, A_{out} = 3.4956 \text{ m}^2, A_{avg} = 3.3204 \text{ m}^2$$

$$\text{Then, } \dot{Q}_r = \sigma A (T_1^4 - T_2^4) \times \frac{\epsilon_1 \epsilon_2}{\epsilon_1 + \epsilon_2 - \epsilon_1 \epsilon_2}$$

$$= 167 \text{ W}$$

Case (II) : Polished outer wall of inner chamber with copper

Now, $\epsilon_{Cu} = 0.02$.

This gives $\dot{Q}_r = 28.09 \text{ W}$

N.B.: Mark the huge difference between the heat inflow when the surface is polished.

(ii) Radiant heat inflow down the sample chamber (SS tube):

This is the radiant heat reaching the inner chamber by funnelling of room temperature.

An upper limit for this value can be calculated by assuming complete funnelling i.e. perfect specular reflection from the inner wall of the tube, so that all the radiation reaches the inner space.

So here $\epsilon = 1$, $A_{SC} \text{ (inside vessel)} = 0.2231 \text{ m}^2$

So, $\dot{Q}_f(\text{max}) = 102.02 \text{ W}$

(iii) Heat conducted down the sample chamber:

$\Delta T = 223$, $K_{avg} = 12.3 \text{ Wm}^{-1}\text{K}^{-1}$, $t_{SC} = 0.0005 \text{ m}$

Cross section area $= 2\pi * r * t = 0.000078 \text{ m}^2$, Length $= 1.3962 \text{ m}$

So $\dot{Q}_c = K_{avg} \frac{A}{l} \Delta T = 0.1532 \text{ W}$

(iv) Radiant heat flow down the transfer siphon:

$A_{TS} = 0.119 \text{ m}^2$

So, $\dot{Q}_f = 51.17 \text{ W}$

(v) Heat conducted down the transfer siphon:

Generally $t_{TS} = 0.0002 \text{ m}$, Cross sectional area $= 0.000016 \text{ m}^2$, $l_{TS} = 1.3962 \text{ m}$

So, $\dot{Q}_c = 0.031 \text{ W}$

(vi) Radiant inflow down the level detector:

$A_{LD} = 0.0839 \text{ m}^2$

So, $\dot{Q}_f = 38.36 \text{ W}$

(vii) Heat conducted down the Level Detector:

$$t_{TS} = 0.0002 \text{ m, cross sectional area} = 0.000012 \text{ m}^2, l_{LD} = 1.3962 \text{ m}$$

$$\text{So, } \dot{Q}_c = 0.023 \text{ W}$$

(viii) Heat conducted down the neck of the inner wall

$$t_{in} = 0.0005 \text{ m, } l_{in} = 1.4962 \text{ m}$$

$$\text{So, } \dot{Q}_c = 1.65 \text{ W}$$

4.3 Stay time for liquid:

The latent heat of N_2 at 77 K at 1atm is 160 kJ/ltr.

The total heat inflow after above calculations comes out to be,

$$\dot{Q}_T(\text{unpolished}) = 360.4072 \text{ W} \quad \& \quad \dot{Q}_T(\text{polished}) = 221.4972 \text{ W}$$

Stay time is given by the ratio of the latent heat to the total heat going into the inner chamber.

Hence 1 litre of N_2 will evaporate in,

443.94 seconds (unpolished surface) & 722.36 seconds (Polished surface)

5 - RESULT & DISCUSSION

- The stay time for one litre of liquid was calculated to be 443.9 secs when the outer wall of inner chamber remains unpolished. But for the polished inner chamber the value was 722.36 secs. So this implies that such an arrangement of liquefying unit cum measurement cryostat is quite feasible. This is an encouraging result.
- The fact that I've always used the higher side of values means the stay time can't be less than this. Also when the baffles & insulation layers are used, the radiant flow decreases drastically. We got an instance of that when we calculated the value for unpolished & polished surfaces. Hence the stay time will only get better than this.
- The cylindrical volume of the inner vessel is 248.2 litres. So once the vessel is completely filled, the liquid will stay for 50 hours when the surface is polished. The present CCR which has been referred here is produces 40 litres per day in optimum conditions. So a CCR of higher cooling capacity is to be installed for a better performance of the system. Now-a-days, a wide range of such refrigerators are available in the industry.

6 - FUTURE WORK

This work has been carried out in a very crude & simple way. So there is a plenty of room for improvement. A thorough simulative study will be worthwhile for optimisation. And fabrication should be achievable with in-depth mechanical & thermal analysis of the arrangement.

We should always hope for the best!

7 - CONCLUSION:

- An attempt was made to design an integrated system of liquefier & a measurement cryostat.
- The materials taken in for considerations were stainless steel & copper. SS was used when we needed strength & low thermal conductivity, whereas copper used in places where high thermal conductivity is needed.
- ASME standards were followed in calculating the thickness of the vessel.
- Dassault Systems' 3D CAD program SolidWorks 2012 was used to make the sample solid model.
- Staying time calculated was quite reasonable which means that such kind of system is possible. They can be used for dual purpose both for doing measurement as well as transferring once they are completely filled.
- This project helped me in getting a feel & flavour of engineering approach followed to make things tangible.

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