Helium Purification by Gas Adsorption method

A Thesis Submitted in Partial Fulfilment of the Requirements for the Degree of

Master of Technology

in

Mechanical Engineering

By

Jitendra Bhushan

Department of Mechanical Engineering

National Institute of Technology

Rourkela

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Under The Guidance of

Prof. Ranjit K. Sahoo

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NATIONAL INSTITUTE OF TECHNOLOGY

ROURKELA

CERTIFICATE

This is to certify that the project review entitled "**Helium Purification by Gas Adsorption method**", being submitted by **Jitendra Bhushan**, in partial fulfillment of the requirements for the award of Master of Technology Degree in **Mechanical Engineering** with specialization in Thermal Engineering at the National Institute of Technology, Rourkela (Deemed University) is an authentic work carried out by him under my supervision and guidance.

To the best of my knowledge, the matter embodied in the thesis has not been submitted to any other University/ Institute for the award of any degree or diploma.

DATE:

Prof. R.K. Sahoo

Department of Mechanical Engineering

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Rourkela – 769008

CONTENTS

ACKNOWLEDGMENT

Setting an endeavor may not always be an easy task, obstacles are bound to come in this way & when this happens, help is welcome & needless to say without help of the those people whom I am going to address here, this endeavor could not have been successful & I owe my deep sense of gratitude & warm regards to my Supervisor **Dr. Ranjit Kumar Sahoo** Professor, Mechanical Engineering Department , NIT Rourkela for his in depth supervision and guidance, constant encouragement and co-operative attitude for bringing out this project work.

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I want to convey my heartiest gratitude to my parents for their unfathomable encouragement. I would also like express heartiest feelings to my brothers and sister in law for providing me encouragement and financial support for higher education. The sacrifice they made to make my dream come true beyond any words I can write.

ABSTRACT

In a cryogenic establishment, helium gas is an expensive consumable. Conservation of helium is important not only for saving on cost, but also for ensuring supply at the time of need. During laboratory experiment, helium gas is often contaminated with air impurities (nitrogen, oxygen, argon, moisture etc.) which must be removed before the spent gas is reused for liquefaction. Therefore, a helium purifier is an integral part of any cryogenic establishment to conserve helium gas by providing grade 4.5 helium or 99.995% pure helium to liquefier after separating air contaminants from impure helium.

The helium purification is based on two principles, one is cryocondensation of moisture and air impurities, on heat exchangers at appropriate temperature and other one is cryosorption on activated charcoal to yield grade 4.5 helium from 60% pure helium at LN_2 temperature and at high pressure of about 150 bar. The purifier has been designed for purifying impure helium upto 40% of impurity by running 6 hours non-stop operation with the mass flow rate of 20 nm^3/hr and delivery of impure helium to purifier at a pressure of 150 bar(a) ,which is ensured by a 3-stage reciprocating compressor. In the helium purifier for cryocondensation and cryosorption process the components are moisture collector vessel, three heat exchangers, liquid air separator vessel and adsorber columns. Other major components are gas bag, compressor, LN_2 vessel and cylinder manifold. Where, all cryogenic components are housed in Superinsulated LN_2 vessel. Purification of helium involved two phase, one is regeneration phase and second is purification phase. For complete removal of moisture from charcoal beds regeneration should be done before purification, by heating and evacuation with purging of pure helium to whole system. And in purification phase we have taken 95% of pure helium and 5% of impurity i.e. dry nitrogen contaminants. Experiment was carried out for one session and four samples are taken in sample cylinder at different interval of time. Sample analysis by Linde Multi Component Detector reveals that the total impurity, consisting of nitrogen, oxygen and moisture is less than 5 ppm be volume, thus making the purified gas much better than grade 4.5 helium.

LIST OF FIGURES

LIST OF TABLES

Chapter 1

INTRODUCTION

Chapter 1

INTRODUCTION

Helium is classified as rare gas, and it is one of the important gases now-a-days, which is becoming a most useful gas in many areas like space research, energy programs and defence as well as the medical, computer and fibre optics field. Against this background the worldwide consumption of pure helium has increased by between 5 to 10 percent a year in the past decade, with biggest growth in its use as a coolant for the superconducting magnets in magnetic resonance imaging (MRI) body scanners. Current helium consumption is estimated to be about 100 million cubic meters, and is predicted to continue to rise by 4 to 5 percent annually. In India, helium is an expensive and exported consumable. Therefore, a helium purifier is an integral part of any cryogenic centre to conserve helium gas.

1.1 Basic principles of helium purification

The basic principle of helium purification is removal of impurities like moisture, air, nitrogen by condensation on different types of heat exchanger passing through it and by adsorption process over activated charcoal. And for cryosorption process helium purifier should works on high pressure and low temperature. For high pressure 3-stage reciprocating helium compressor and for low temperature LN2 cylinder is used which is filled with liquid nitrogen at 77K. Impure helium gas contains some contaminants like lubricating oil of compressor which can be removed in moisture separator vessel. From pure helium cylinder and from nitrogen cylinder, both pure helium and nitrogen are mixed, where gas bag collects this impure helium and supplied to compressor inlet at low pressure. The compressor compressed impure helium to 120 bar or upto150 bar and discharge into helium purifier LN2 cylinder where this high pressure reduces the fraction of impurities at different stages before it enter into adsorber columns. After compressor, impure helium gas passes through moisture separator vessel, three heat exchangers ,liquid air separator vessel and the remaining (0.83%) impurity enters into charcoal adsorber columns, where air impurity reduces to 50 ppm and thus yield 4.5 grade pure helium, which can be checked in linde multi-component detector. The helium purifier works on

two phases, one is purification another is regeneration. After every cycle of purification phase regeneration should be done to remove moisture by heating and evacuation.

1.2 Objective of the study

At present scenario, pure helium is used in many applications at different organizations and research centres in India. But, it is not possible to import helium purifier for every organization because it is very expensive and costly to maintain. Purification of impure helium by indigenous developed purifier will make it easy to get pure helium at low cost. The objective of the work is as follows:

(i) Testing and commissioning of helium purifier based on cryosorption and performance analysis under varying input parameters.

(ii) Proper documentations for future study on purification with various impurity concentrations.

(iii) To obtain pure helium of 4.5 grade or 99.995% ppm by gas adsorption process using coconut shell granular activated charcoal as adsorbent.

1.3 Principal components of helium purification process

The components of helium purifier are:

1. Compressor
2. Purifier LN2 vessel with top flange
3. Adsorber columns
4. Tubular heaters
5. Back pressure regulator
6. Gas bag
7. Oil and moisture separator vessel
8. Liquid air separator vessel
9. Heat exchangers (tube-in-tube, shell and tube, helical coiled)
10. Helium purity monitor
11. Pure and impure helium manifold

Chapter 2

LITERAURE REVIEW

Chapter2

LITERAURE REVIEW

Cryogenic technology is the study of production of very low temperature (below –150 ℃, or 123 K) and the behaviour of materials at those temperatures. For the purification process, development of such low temperature working device, air separation and fundamental principles and procedures have been discussed in well-known text books of cryogenics [1-8].For the basic concept of cryogenic and low temperature adsorption systems the books [1][2][3][8] gives introductory knowledge in this field.

2.1 Idea behind Gas Purification System

Linde Group developed the world's first air separation plant for the production of oxygen in 1902 and the first production facility was set up as early as 1903 in Hollriegelskreuth near Munich. In 1989, the linde group installed the largest pressure swing adsorption plant in Europe with vacuum regeneration for production of oxygen from air [26].Line North America is the one of the world's largest helium supplier and since 1994 linde has been the sole provider of helium[26]**.**

2.2 History of Helium

1868 is the year that marked the beginning of the helium story [26].Independently of each other, a French astronomer, Pierre Jules Cesar Janseen, and an English astronomer, Sir Joseph Lockyer, simultaneously identified a previously unknown element in the sun's spectrum. And then Lockyer named this new element as Helium. However, it was not until 1895 that the existence of this new element could be proven, when Sir William Ramsey, a Scottish chemist, discovered helium in uranium minerals on earth and later in atmosphere. In 1903 helium was fond in natural gas which has remained the main source of helium since [26]. Helium is colourless, odourless, non-toxic, non-corrosive and non-combustible. This all properties of helium are provided in the book of thermodynamic property of cryogenic fluid by Richard, Steven and Eric [6].With a value of 4.2 degree Kelvin or 296^{o}C, it has the lowest boiling point among all the gases and as a result Liquid Helium is the coldest matter on Earth[26]. This is what makes it ideally

suited to be used as cryogen in a number of cutting edge applications including superconductivity in magnetic resonance imaging (MRI), nuclear magnetic resonance (NMR), particle physics and other cryogenic applications. As a gas the special properties of helium are used in many industries and processes such as diving, lifting, leak testing, automotive industry, semiconductor manufacturing, cutting and welding, nanotechnology and for analytical purposes.

2.3 Helium purification by cryocondensation and cryosorption process

Purification of helium is possible by multi-stage adsorption process. For purification of any gas there are multiple process with various engineering techniques which are well described in Barron [1] and kohl, risenfield [17].In many cases, the separation of mixture at cryogenic temperatures is the most economical separation method of all.sPractically all the commercially produced oxygen, nitrogen, argon, neon, krypton and xenon are obtained through rectification of liquid air .Other separation method, such as physical adsorption and refrigeration purification, are increased in effectiveness as the temperature is lowered into the cryogenic range. Kinday and Hiza[13] gives the thorough description of physical adsorption in cryogenics. Also Basmadjian[14] addresses the basic adsorption phenomenon and provides an idea about absorber bed sizing, estimation of bed and purge requirement. Perry's handbook [22] is a very effective tool for selection of adsorbent. Values of adsorption capacity of granular activated and compressed activated carbons were determined by BET method and by liquid nitrogen adsorption followed by extrapolation method have been presented. Kidney et. al. [12] have presented pure component and binary adsorption isotherms for nitrogen adsorption on coconut shell charcoal at LN_2 temperature and over pressures ranging from atmospheric to 80 bar(g).

Stoll et. al. [25] commissioned a fully automatic large capacity helium purifier with operating at 150 bar(g) pressure, volumetric flow rate 135 nm^3/hr (maximum), output air impurity level of less than 10 ppm, with cycle time of 24 hours which included operation, regeneration, re-cooling and re-pressurization time . The adsorption bed was silica gel dipped in LN_2. Two heat exchangers, one immersed in the LN_2 bath and the other operating at 65 K existed ahead of adsorber bed. Regeneration of the adsorber bed was carried out by heating the adsorbent to 150 K, pumping out desorbed impurities, back

filling with helium, re-pumping and finally backfilling with pure helium. This purifier unit was equipped with two adsorber beds, one running and other stand by.

In VECC, Kolkata, India [15] already a small model is tested. It was tested with impure helium at pressure of 20 bar(g), flow rate of 1.6 nm^3/hr and achieved helium purity of 99.99% from about 1% air and moisture impurity. Activated charcoal bed at LN_2 temperature was used for adsorption process.

Thingstad [24] have explained briefly about the cryogenic purifier installed in streamer chamber at Stanford Linear Accelerator Centre, USA. This purifier was used for purifying neon-helium mixture with impurities nitrogen, oxygen and moisture. Impure gas was pumped through moisture dryer containing Linde-type 13X molecular sieve, following which there was a heat exchanger and cryogenic adsorber unit operating at LN_2 temperature. The dryer and adsorbent were regenerated by baking at 260^oC for two hours, the last half-hour under vacuum.

In India, a project [9] on extraction of helium from natural gas was taken up by the Department of Atomic Energy and Department of Science and Technology. Here they applied pressure swing adsorption process in the absence of any cryogen, a constant stream of helium with purity greater than 99% by volume was obtained. The plant intake was constant at 50 nm^3/hr of natural gas containing 0.06% by volume of helium operating in the pressure range of 4 to 5 bar (absolute). This plant was a four stage adsorber based pilot plant with complete automation and microprocessor based logic controllers. The adsorbents used in various stages were silica gel, activated carbon and zeolite.

Chapter 3

EXPERIMENTAL DESCRIPTION OF PRINCIPAL COMPONENTS OF PURIFIER

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Helium purifier which works on condensation and cryosorption process yields 99.995% pure helium. For purification of helium some input parameters are necessarily maintained though the helium purifier is designed upto that value. Experimental set-up is shown in P & I diagram, which is shown in fig. 3.1.

3.1 Technical Specification of Helium Purifier

Flow rate	: 20 nm^3/hr.
Operating pressure	: 120 bar
Operating temperature	: LN_2 temperature i.e. 77K
Input gas purity	: 60% i.e. 40% air impurity (maximum)
Output gas purity	: 99.995% (minimum) i.e. 4.5 grade, i.e. max. allowable impurity is 50 ppm by volume
Run time for purification	: 6(six) hours
Regeneration time	: 4(four) hours
Regeneration process	: Heating and evacuation
Regeneration temperature	: 120^oC
Type of heater	: Ni-Chrome element based tubular heater
Adsorbent	: Coconut shell granular activated charcoal

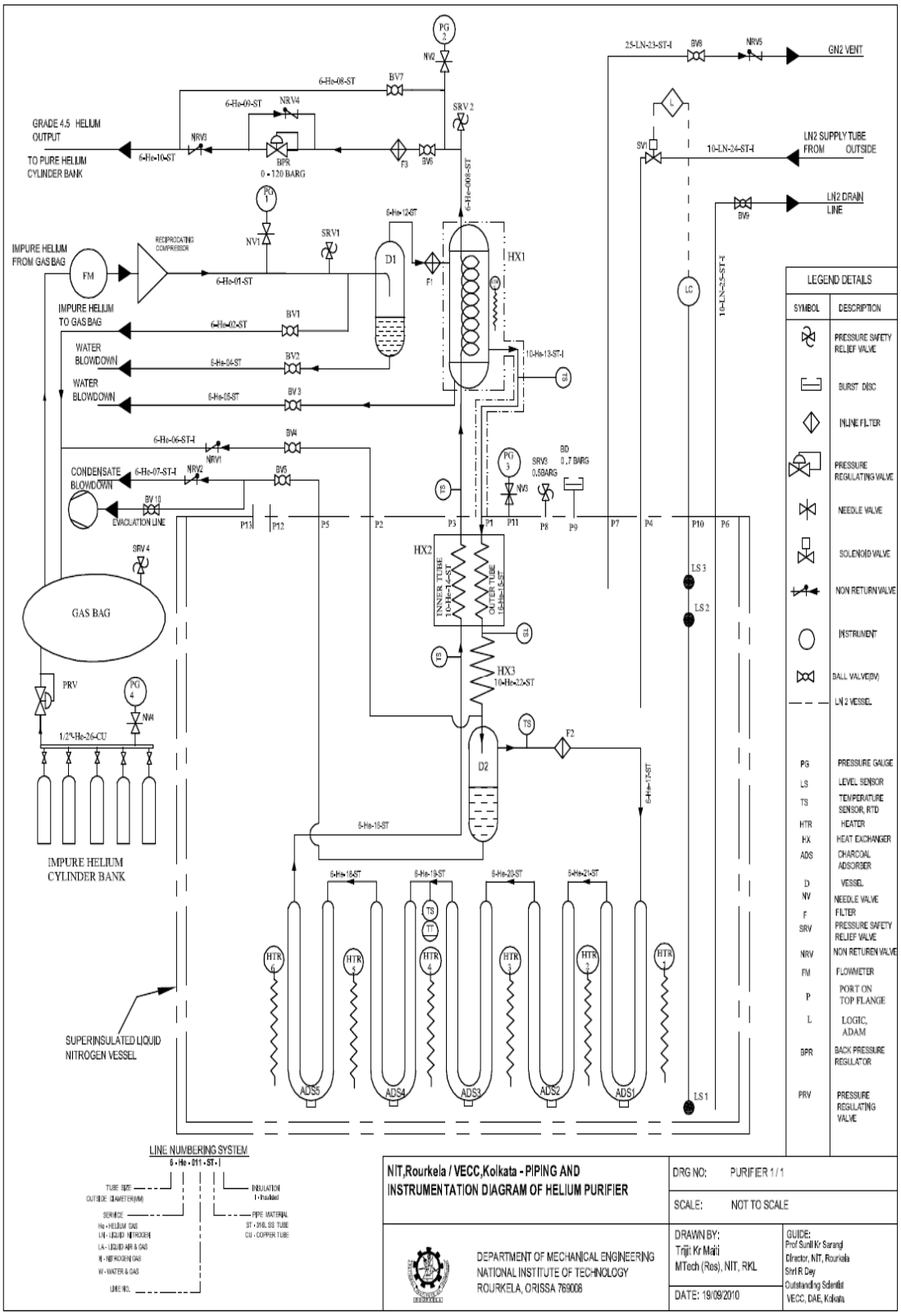

Fig.3.1: P & I Diagram of Helium Purifier

3.2 Detailed process of components of purifier

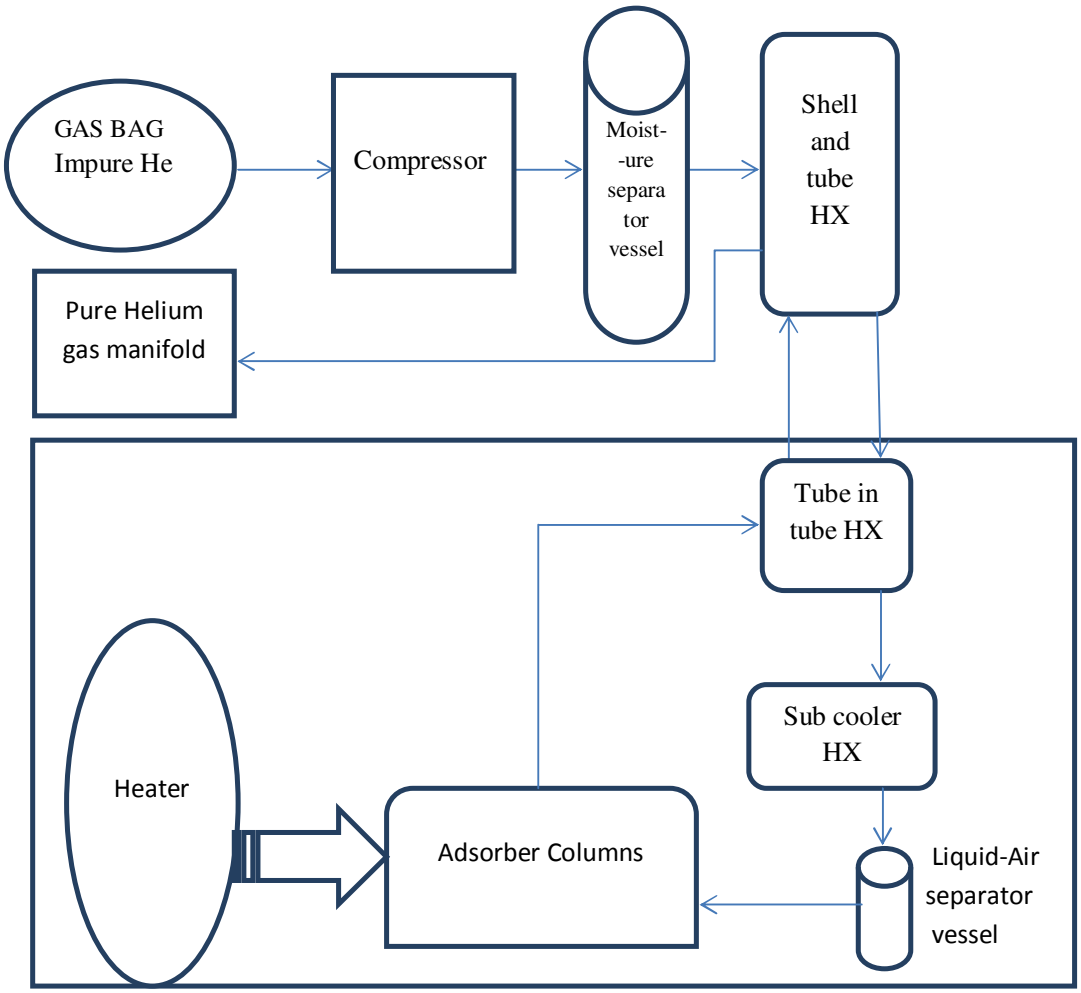

Fig.3.2: Block diagram of helium purifier system

Basic purification process

For purification of helium we come across two phases:

- Purification phase
- Regeneration phase

Purification Phase

- In first phase i.e. Purification Phase, the impure helium gas from impure manifold which is at 150-200 bar is send to gas bag through pressure regulating valve. The

operating pressure of gas bag is 50 mbar gauge which is close to atmospheric pressure.

- Impure gas from gas bag is compressed to 120-150 bar by using high pressure three stage reciprocating helium compressor and delivers to purifier where due to high pressure moisture and oil are settled down in moisture separator vessel.
- After passing moisture separator vessel it enters into Shell and tube heat exchanger where major part of moisture gets removed and then passes to second heat exchanger i.e. tube in tube type which is placed inside LN2 vessel. The moisture free gas enters in tube in tube heat exchanger where it is precooled by purified helium gas coming out from adsorber columns which is fully submerged in LN2 at 77K and at low temperature most of nitrogen and methane condense into liquid and drained off.
- The third heat exchanger subcooler coiled heat exchanger is completely submerged in LN2.By passing impure helium through this air gets liquefied and at liquid air separator vessel, the condensed liquid air will get separated and will be drained off by opening valve.
- The remaining impurity gets purified by adsorber bed of activated charcoal and 99.995% pure helium will be obtained which is verified by linde multi-detector device.

Regeneration Phase

- After running purification for six hours, the adsorber beds are depleted of adsorption capacity by an increase in contaminants level and moisture. After purification phase it is necessary to regenerate the adsorber columns and whole piping networks.
- Efficiency of purifier depends on effectiveness of the regeneration process.
- Regeneration process follows:
    - First the system will be depressurized and liquid nitrogen will be removed from LN2 vessel.
    - Six heaters are provided for heating process each of 1000w.the heater heated the adsorber columns and the piping at 120^{o}c.
    - At the time of heating evacuation is done by vacuum pump for desorption process on charcoal and then slow reverse flow of pure helium is given for purging and back refilling.
    - On completion of regeneration the system is completely purged with grade “A” helium and then after only it is ready as it is confirmed that there is no impurities or moisture, air remains inside purifier and charcoal.

3.3 Description of components:

Gas bag

This gas bag is made up of four layers of different materials of rubberised fabric with low permeability of helium and this is designed for the purpose of collecting impure helium, which comes from impure manifold or experimental set up. It operates at a pressure of 50 mbar (g) and acts as buffer at the compressor inlet. The only media of impure helium to compressor is from gas bag at low pressure.

Compressor

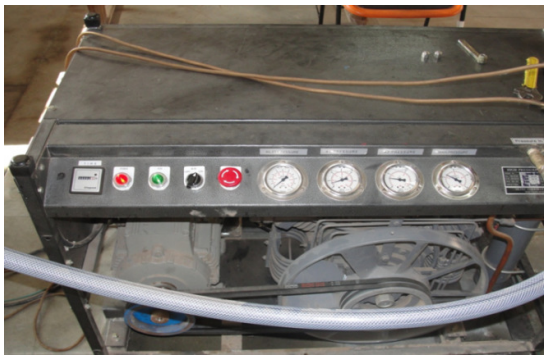

Fig.3.3: 3-Stage Reciprocating Helium Compressor

A special type of compressor is used, which is 3-stage reciprocating type high pressure helium compressor. From gas bag, impure helium comes to compressor inlet at nearly atmospheric pressure and then compresses to 150 bar pressure and delivers to purifier at a flow rate of 20 m^3/hr. This compressor is specially made for helium which is completely sealed and has very low helium leak in the order of $10^{-4}mbar^{-1}/s$. The discharging pressure starts with the minimum pressure at which impure helium is supplied at compressor inlet and rises as our requirement with respect to time.

Moisture Separator Vessel

After compressor, impure helium delivers to purifier at high pressure of 150 bar to this vessel, which has ability to accumulate condensed moisture and oil which comes from compressor. As the compressor is lubricated with oil, so at the outlet of the compressor impure helium comes with moisture and oil which should be removed in this vessel. And blow down is done at regular intervals to remove this impurity. Otherwise, after sometime

moisture separator vessel will be full of impurities (moisture and oil) and it will enter to purifier which is not condensate free gas.

Fig.3.4: moisture separator vessel

Shell and Tube Heat Exchanger

Fig.3.5: shell and tube heat exchanger inside which coil is bundled

It is a counter current heat exchanger in which coiled tube is bundled inside the shell. In this heat exchanger major part of moisture will get removed and the condensate will be removed periodically by manual drain valve. Impure helium at normal temperature flows inside the shell over the coiled tube and through tube, cold pure helium flows. The moisture gets condensed on the tube surface and is collected at the bottom of the shell.

Tube in Tube Heat Exchanger

A typical double-pipe or tube in tube heat transfer exchanger consists of one pipe placed concentrically inside another pipe of a larger diameter with appropriate fitting to direct the flow from one section to the next. Due to the reasons of high efficiency, compactness, easy to fabricate this type of heat exchanger has been selected as cryogenic heat exchanger. This is tube-in-tube helically coiled counter current flow heat exchanger made of 16 mm OD and 10 mm OD SS316 tubes. Pure helium gas at 80 K enters heat exchanger from inside tube and exits at 290 K when

impure helium enters at 300k from outer tube of heat exchanger and getting cooled to around 100K with heat exchanger effectiveness of 80%.

Fig.3.6: Tube in Tube heat exchanger

Subcooler Heat exchanger

Fig.3.7: Subcooler heat exchanger

This heat exchanger is helically coiled and it is fully submerged in LN2 with the LN2 vessel. This mainly works as a subcooler, in which nitrogen impurities gets condensed and collected in liquid air separator vessel which is just below the subcooler heat exchanger.

Liquid Air Separator Vessel

It is situated inside the LN2 vessel and completely submerged with LN2. This vessel acts as phase separator where condensed air from purifier feed impure helium gets collected. The feed stream enters tangentially and rotates in cyclonic motion inside the vessel for efficient separation of liquid phase from gas stream and is collected at bottom due to gravity. After liquid air separator vessel only 0.83% impurity is allowed to enter into adsorber columns. The helium gas escapes through the port at the top of the vessel. The condensate is blown down periodically.

Adsorber Columns

Adsorber bed consists of five U-shaped vertical stainless steel columns connected in series. Inside this columns coconut shell granular activated charcoal is packed for adsorption process. This columns are fully submerged in LN2 and hence at 77K. Adsorption of air and moisture at 77K and at high pressure of about 120 bar which is maintained by back pressure regulator ensured that inside columns pressure is 120 bar. Anthracite is most common adsorbent in helium purifier but due to large BET surface area about 1600 m^2/gm coconut shell based charcoal is used in the purifier for adsorption process. At both the ends of U shaped vessel, stainless steel wire mesh filter attachment is welded to filter out the charcoal dust from the outgoing pure helium.

Tubular Heater

Fig.3.8: Arrangement of Tubular heaters

Ni-chrome wire based tubular heaters are used to heat up the adsorber bed, heat exchangers, snow filter and liquid air separator vessel during regeneration process. Six heaters and adsorber columns are arranged in such a manner to heat up adsorber columns uniformly at 120^{o}C.Desorption of impurities takes place from adsorber and condensed air, moisture gets evaporated from liquid air separator vessel. The evaporated air and moisture are sucked by vacuum pump during regeneration process.

Back Pressure Regulator

Back pressure regulator controls upstream pressure or back pressure at the delivery port where pure helium comes out from adsorber columns. It is very important to maintained

high pressure of about 120 bar inside the adsorber columns and purifier system to remove impurities. This regulator maintains the pressure of 120 bar to adsorber bed during purification stage by externally adjustable relief valve and ensures most efficient adsorption at particular pressure.

LN2 Vessel of Purifier

Fig 3.9: LN_2 vessel with flange and without flange

This is a Superinsulated LN2 vessel, which is insulated by high vacuum in order of 10^3.All the cryogenic components are houses in LN2 vessel starting from heat exchangers, heaters, liquid air separator vessel, LN2 level indicator and at the bottom adsorber columns are placed in series. This consists of two parts: top flange and vessel. The bolted top flange seals the LN_2 vessel with an O-ring. The internal components of the purifier have been placed in a cage which is suspended from top flange. At the time of operation either purification or regeneration only from the top flange one can perform process.

Pure and Impure Cylinder manifold

These are high pressure storage system for both pure and impure helium gas. In pure manifold gas is collected at high pressure at 120 bar from purifier. And impure gas can be collected from various sources viz. cryogenic experimental set up, helium liquefier at various modes of operation and by directly pure helium cylinder by mixing some percentage volume of nitrogen and air. From impure manifold gas is sent to gas bag where it stored impure gas and deliver to compressor.

Chapter 4

DETAILED DEVELOPMENT AND THEORITICAL ANALYSIS OF PERFORMANCE OF PURIFIER COMPONENTS

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DETAILED DEVELOPMENT AND THEORITICAL ANALYSIS OF PERFORMANCE OF PURIFIER COMPONENTS

4.1 Design and development of helium gas bag

For temporary storage of impure helium gas at low pressure close to atmospheric pressure a gas bag is prepared by nit Rourkela in collaboration with M/s Softex Industrial Products Pvt. Ltd, Kolkata, India. Presently, liquid helium user establishments of India, import gas bags which are very expensive in terms of foreign exchange and long delivery items but also difficult to repair minor damages.

The gas bag is mainly multi-layered rubber bag and it has following properties: This gas bag material is weathering resistant, fungal resistance and corrosion resistant to most organic solvents like ketones, alcohols, gasoline, oil, etc and weathering resistant. It also has excellent retention of physical and chemical properties at very low temperatures including cold abrasion resistance etc. We faced some problems with nozzle of this sample. The nozzle was made of SS304 pipe which was interfaced with gas bag material using adhesive, this interface was leaking at operating pressure. So, we redesigned the nozzle for 5 cubic meter gas bag and the system is working nicely.

Fig.4.1. Gas Bag installed at NIT, Rourkela

4.1.1 Technical details of helium gas bag

The technical specification of the gas bag is as follows:

- Volume: 5 cubic meters. Length of gas bag is 3 meters, width (deflated condition) 2.5 meters and diameter (inflated condition) 1.6 meter. Total weight is 18 Kg.
- The gas bag material is textile reinforced rubber having 4 layers viz. chloro-sulphonated polyethylene rubber, polyamide cloth, polychloroprene rubber and polyester cloth, thickness being 0.40 mm, 0.30 mm, 0.20 mm, 0.20 mm respectively and cloth thickness is 1.0 ± 0.10 mm.
- Physical properties of cloth are as follows:
    - Breaking Strength (kg/20mmwidth)
      Warp > 1000, Weft > 1300
    - Elongation at break (%,20 mm width)
      Warp > 20, Weft > 20
    - Tear Strength (kg/20 mm width)
      Warp > 200, Weft > 200
    - Weight : 1100g/m^2
- Working pressure: 50 mbar (0.75 psi)
- Test pressure 100 mbar as per standard.
- Width of the cloth used for gas bag: 1.28 m or more
- Two hollow cloth loops of proper size for 25 mm NB pipe on each side of gas bag, and one loop at the top to support float for limit switch, i.e., total of 5 loops are there The weight of float is about 300 gm in weight.
- As nozzle area is the most critical zone, so proper precautions have been taken during its fabrication. Nozzle size: 25 mm, Nozzle number: 1(one) nos. The flange material of nozzle is Teflon and is injection molded class 150 flange. Two flanges, one inside gas bag and other outside. The bolts used in flange are stainless steel (AISI 304) and size M10. The extended pipe is clamped with PVC braided hose of 25 mm inner diameter. Neoprene rubber washer is used between flange and cloth both inside and outside for sealing. The inside flange is 25 mm thick and have tap depth of at least 15 mm.
- This gas bag is suspended from ceiling by mild steel structure. Mild Steel pipes of 1” NB, light weight, are anchored to ceiling by anchor bolts. Gas bag has two

loops on each side lengthwise for fastening with Mild Steel structure.Fig.4.1 shows gas bag installation. Fig 4.2 describes the details of the nozzle of gas bag which is a very crucial part of the system. This part has been designed carefully as it is very vulnerable part for leakage.

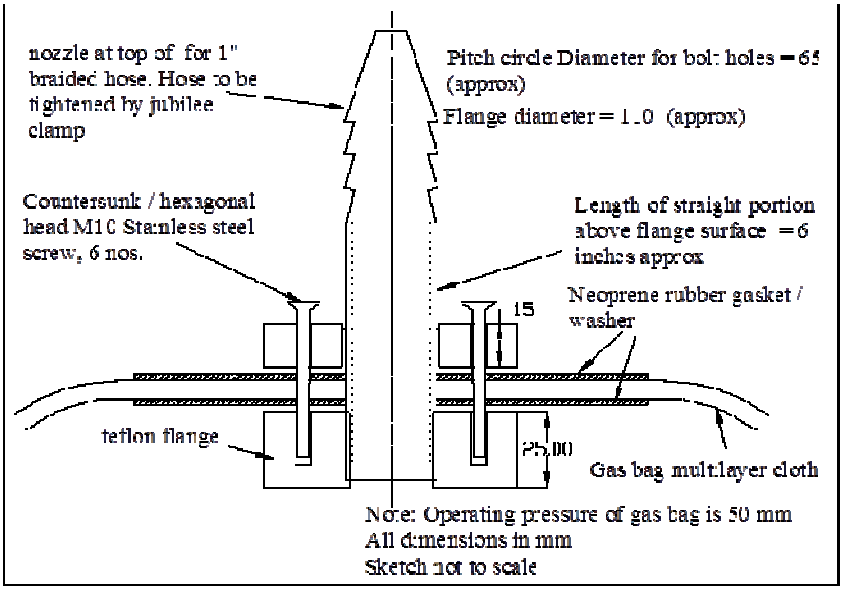

Fig.4.2 Nozzle of the Flange of Gas Bag

4.1.2 Test at manufacturer's site

Initially, gas bag was evacuated for half an hour by rotary vacuum pump for removal of moisture and air. Then dry compressed air was filled to raise pressure up to 200 mbar(g) at 34^{o}C ambient temperature. Gas bag pressure was observed hourly with readings of both pressure as well as temperature noted. Gas pressure was measured by bourdon gauge with the least count of 0.50 mbar. After 79 hours, the pressure drop was found to be 2.50 mbar at same ambient temperature. The pressure drop was quite negligible, so the product was satisfactory and acceptable.

4.2 High Pressure Helium Compressor Selection

Selection of compressor is very important because what we need is high pressure of about 150 bar (g) to ensure efficient condensation and adsorption of impurities and hence we will obtain better adsorption isotherm. A truly indigenous compressor with the required flow rate, pressure rating and helium leak tightness is not available in Indian

market, but imported ones are readily available. The compressor we used here is from Metec Corporation, South Korea and its technical specifications are:

- Flow capacity: 21 Nm^3/hr
- Inlet gas pressure: less than 0.5 bar(g)
- Operating pressure: 150 Bar(g) [210 bar(g) (maximum)]
- Pressure switch setting pressure: 150 bar(g)
- Safety valve setting pressure: 210 bar(g)
- Discharge valve outlet port: ¼”
- Oil removal unit consists of two micron filters in series viz. 1 micron and 0.01 micron with auto drain valve. An activated charcoal filter is there at the end for final removal of oil vapor.
- This high pressure helium gas booster compressor is of make M/s Metec Corporation, Korea which is cheaper than any USA or European model. Our model costs Rs. 10 Lakhs.

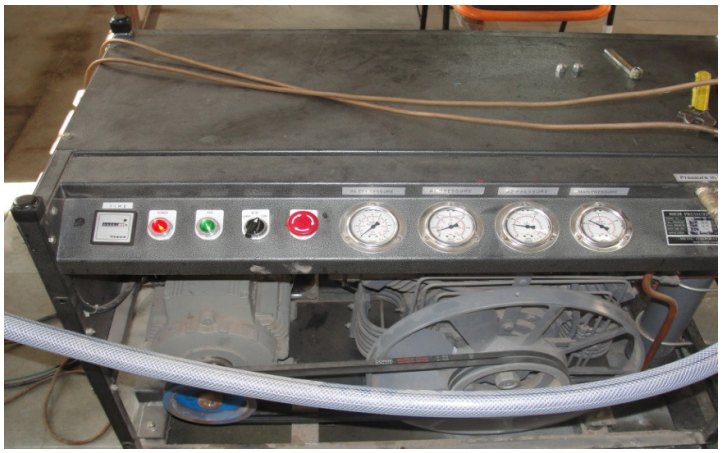

Fig.4.3.High Pressure helium compressor

In India, all the helium users are presently importing compressors and its spares at very high cost in terms of foreign exchange. The modest effort of NIT, Rourkela will help the institutions to utilize a much cheaper version of helium compressor in near future. So, after several operational runs of helium purifier we will be able to draw the detailed conclusion about the performance of this compressor

4.3 Design and development of Activated Charcoal adsorber columns

Activated charcoal, the heart of purifier system which removes all traces of impurities from impure helium at cryogenic temperature by cryosorption. Granular activated charcoal is the best one having maximum BET surface area and less bulk density as compared with the adsorbents commonly available in Indian market are Activated Alumina, Molecular Sieve or Zeolite, Silica Gel and Activated Charcoal of wood based, lignite based, peat based, bituminous coal based, anthracite coal based, petroleum based, etc. More BET surface area means more micro-pores for multi-layered gas adsorption. The lesser bulk density of activated charcoal reduces the adsorber bed weight. Its regeneration temperature is also lower than other adsorbents i.e. it is much easier and quicker to reactivate the saturated charcoal.Nitrogen adsorption isotherm [13][21] of activated charcoal is better than molecular sieve, which are commercially available like, silica gel and activated alumina at cryogenic temperature and higher pressure. Also, granular activated charcoal has better adsorption property than its pellet form at low temperature.

India is one of the major producers and exporters of coconut shell charcoal in the world and this is quite cheaper in India.

Fig 4.4: Coconut shell granular activated charcoal

Hence, coconut shell granular activated charcoal has been chosen in preference to other adsorbents for helium purifier. The indigenous activated charcoal used for this work has been purchased from M/s Exal Corporation, Vadodara, Gujarat, India. The typical characteristic which has been available to us from the manufacturer is shown below in Table 3.2. BET surface area has been measured in the institute.

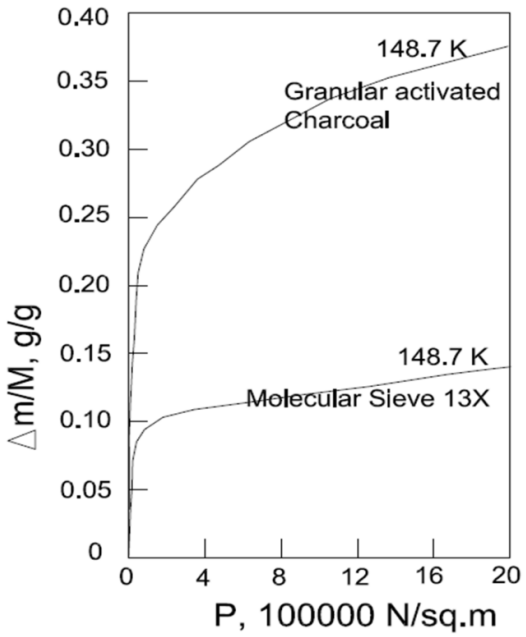

Fig.4.5: Nitrogen adsorption isotherms on molecular sieve and activated charcoal.

Table 4.1: Technical Data Sheet of Activated Charcoal Grade AC 4/8

Characteristics	**Property of charcoal**
Make	M/s Exal Corporation, Vadodara, Gujarat, India
Grade	AC 4/8
Raw material	Coconut shell
Particle type	Granular
Sieve size	- 4 + 8 BSS
BET surface area, m^2/gm,(min)	1600
Moisture, percent by mass (min)	4.5
Ash, percent by mass (max)	4.0
Adsorption capacity in terms of Iodine number (min), mg/gm	608
Apparent density, gm/cc	0.55

4.3.1 Adsorbent Mass Requirement

Impurity that remains after condensation in tube-in-tube and subcooler heat exchanger will be adsorbed in adsorber columns. After passing from liquid air separator vessel impure helium is saturated with 0.83 % air which will have to be adsorbed by activated charcoal to obtain grade 4.5 helium.

There are two cases for calculating the remaining impurity after liquid air separator vessel at flow rate of 20nm^3/hr and at a pressure of 120 bar minimum.

(1) Impure helium containing 40% air impurity .

(2) Impure helium containing 40% nitrogen impurity.

1) When helium contains 40% air impurity then the remaining percentage of air impurity which delivers to adsorber bed after liquid air separator vessel is calculated here:

   Air contains 78% nitrogen and 22% oxygen

   Saturation pressure of nitrogen is 1 bar and oxygen is 0.2 bar at 77 K.

   According to Raoult's law,

   Partial pressure of nitrogen at 77 K

   = Saturation pressure of N_2 at 77 K x Mole fraction of N_2

   = 1 x 0.78 = 0.78 bar

   Partial pressure of oxygen at 77 K

   = Saturation pressure of O_2 at 77 K x Mole fraction of O_2

   = 0.2 x 0.22 = 0.044 bar

   Partial pressure of air at 77 K after liquid air separator

   = (0.78 + 0.044) bar = 0.824 bar

   Minimum adsorption pressure = 120 bar(a)

   Mole Fraction of air at 77 K

   = (0.824 / 120) x 100

   = 0.69%

Helium containing 0.69% remaining air impurities enters the adsorber columns after liquid air separator vessel.

2) When helium contains 40% nitrogen impurity then the remaining percentage of nitrogen impurity which delivers to adsorber bed after liquid air separator vessel is calculated here:

   Saturation pressure of nitrogen at 77 K is 1 bar

Partial pressure of nitrogen at 77 K = 1 bar

Minimum adsorption pressure = 120 bar(a)

Mole Fraction of nitrogen at 77 K after liquid air separator vessel

= (1 / 120) x 100

= 0.83%

Helium containing 0.83% remaining nitrogen impurity or lower enters the adsorber columns after liquid air separator.

In the second case, helium containing higher impurity concentration enters the adsorber columns, hence the design of the adsorber columns is done based on 0.83% remaining nitrogen impurity in helium at a flow rate of 20 nm3/hr for 6 hr purification process.

Quantity of nitrogen to be adsorbed

= 0.83% of 20 nm3/hr flow rate for 6 hr

= [(0.83 x 20 x 6) / 100] nm3

= 0.996 nm3

= 0.996 x 10^6 cc

Quantity of adsorbent required to adsorbed 0.996×10^6cc of nitrogen impurity:

BET theory aims to explain the physical adsorption of gas molecules on a solid surface and serve as the basis for an important analysis technique for the measurement of the specific surface area of material.

The BET equation of monomolecular layer formed over adsorbent surface is expressed as:

$$\frac{V}{m_a} = \frac{v_m z\left({}^{p}/_{p_{sat}}\right)}{\left(1 - {}^{p}/_{p_{sat}}\right)\left[1 + (z-1)\left({}^{p}/_{p_{sat}}\right)\right]}$$

Where V = volume of gas adsorbed at 101.325 kpa and 273.2 k

m_a = mass of adsorbent

v_m = volume of gas per unit mass of adsorbent required to form a monomolecular-layer over the entire adsorbent surface

p = partial pressure of the gas being adsorbed

p_{sat} = saturation pressure of the gas being adsorbed at the temperature of the adsorbent

The parameter z is a function of energy of adsorption and the temperature of the adsorbent,

$$z = exp\left({\theta_a}/{T}\right)$$

The value of the parameters v_m and θ_a for charcoal adsorbent and nitrogen gas is given by BET theory [1] i.e. v_m= 181.5 cc/gm is the volume of nitrogen needed to form a monomolecular layer of gas over the total adsorbent surface per unit mass of adsorbent when temperature is 77K and θ_a= 300.2

The BET equation may be extended to the case in which only n layers of the gas can be adsorbed,in which the following applies:

$$\frac{V}{m_a} = \frac{v_m z x}{1-z}\left[\frac{1-(n+1)x^n + nx^{n+1}}{1+(z-1)x - zx^{n+1}}\right]$$

Where $x = {p}/{p_{sat}}$

For monomolecular adsorption n = 1, so the equation becomes

$$\frac{V}{m_a} = \frac{v_m z(p/p_{sat})}{1+z(p/p_{sat})}$$

We can find the parameter z:

$$z = exp\left({\theta_a}/{T}\right) = exp\left({300.2}/{77}\right)$$

$$z = 49.338$$

And $V = 0.996 \times 10^6, (p/p_{sat}) = 120$

Therefore, mass of activated charcoal requirement is:

$$m_a = \frac{0.996 \times 10^6[1 + 49.338(120)]}{181.5 \times 49.338 \times 120}$$

$$m_a = \frac{5897873760}{1074581.64}$$

$$m_a = 5488.3\ gm$$

$$m_a = 5.488\ kg$$

This helium purifier system works on thermal regeneration, so estimation of adsorbent requirement is based on 70% saturation of adsorber bed as suggested by Haselden [8]. Hence, quantity of activated charcoal requirement

$$= 5.488/.70$$

$$= 7.84 \text{ kg}$$

Minimum amount of activated charcoal required for helium purifier is 7.84 kg.

4.3.2 Design of Adsorber column

Cross-section of adsorber column:

Velocity of helium stream through adsorber bed should have optimum value, as high value of approach velocity through adsorber bed results in bed fluidization, powdering of charcoal particles and sufficient time for proper adsorption on adsorbent surface.

Approach velocity (V) of gas through fixed adsorber bed ranges between 10^{-2} to 1 m/sec [14]. So the dimensions of bed has been designed to keep gas velocity near 10^{-2} m/sec or lower

Operating pressure of purifier = 150 bar and flow rate = 21 Nm^3/hr.

Therefore, the flow rate of helium at 150 bar(a) and 77 k = $9.44\times10^{-6} m^3$/sec

Flow rate of helium/V = $(\pi/4)\times D_i^2$

$9.44\times10^{-6} = (\pi/4)\times D_i^2\times(10^{-2})$

Therefore, $D_i = 0.03647$ m = 34.67 mm

Hence, inner diameter of adsorber column comes out to be 34.67 mm. So, such pipe should be selected whose inner diameter greater than 34.67 mm and the gas velocity through adsorber bed have optimum value. The adsorber columns will be immersed in LN_2 and will also expose to 120^o C in cycles, so austenitic stainless steel of specification ASTM A 312 TP 316 L as adsorption column pipe material is selected.

Let us select 50 NB and 40 NB pipes

Pressure of gas, P = 150 bar(g)

As per ASME B 31.3 (1999), Process Piping, the allowable internal pressure is calculated by using the following formula:

Allowable internal pressure, $P = (2 \times t_{min} \times S) / [OD - (2 \times Y \times t_{min})]$ ……(i)

Where,

P = 150 bar or 2.205 ksi

Allowable stress,S = 16.7 ksi for SS304

Y = 0.40 for $t_{min} < OD/6$,

Y = $\{OD - (2 \times t_{min})\} / 2(OD - t_{min})$ for $t_{min} = OD/6$

t_{min} = Minimum wall thickness

OD = Nominal outside diameter

Calculation of thickness with 50 NB pipe size

Outer diameter of 50 NB is = 60.30 mm

Applying Eqn. (i), we get

$$2.205 = (2 \times t_{min} \times 16.7) / (60.3 - 2 \times 0.40 \times t_{min})$$

$$t_{min} = 3.78 \text{ mm}$$

So, pipe of size 50 NB Sch 40 (OD = 60.3 mm, t = 3.91 mm) is suitable

Calculation of thickness with 40NB pipe size

Outside diameter of 40 NB pipe is 48.3 mm

Applying Eqn. (i),

$$2.205 = (2 \times t_{min} \times 16.7) / (48.3 - 2 \times 0.40 \times t_{min})$$

$$t_{min} = 3.03 \text{ mm}$$

Pipe of size 40 NB Sch 40 (OD = 48.3 mm, t = 3.68 mm) is suitable.

So, 40 NB Sch40, t_{min}= 3.91mm is suitable for us, but we can choose one higher value of thickness in standard size for safe design due to pressure fluctuation. Hence, ASTM A 312 TP 304 50NB Sch80 has been chosen.

4.3.3 Length Calculation of Adsorber Column

Apparent density of activated charcoal used in purifier (Grade AC 4/8)

$$= 550 \text{ kg/m}^3$$

Let length of adsorber column be 'L' m

Inside diameter of adsorber column (size 50 NB Sch 80), $d_i = 0.04922$ m

Volume of adsorber column

= Volume of activated charcoal of mass 7.84 kg [refer subsection 4.3.1]

Therefore,

$$\pi/4 \times d_i^2 \times L \times \text{apparent density} = \text{Mass of activated charcoal}$$

$$\text{or, } L = (7.84) / \{\pi/4 \times (0.04922)^2 \times 550\}$$

$$= 7.49 \text{ m}$$

Adsorber column is vertical U-shaped, and charcoal content length of each column is assumed to be 2 m. (Assumption is based on the optimisation of dimensions and layout of LN_2 vessel and purifier components)

Number of adsorber columns

= 7.49 / 2

= 3.74

Minimum number of adsorber column required is 4

One more redundant column has been added for enhancing performance and for some unforeseen factors. Total number of adsorber columns becomes 5.

L/D Ratio

Bed lengths of less than 1.30 m, with Length / Diameter ratio less than 1 may suffer from flow maldistribution, as per Haselden [6]. In U-shaped adsorber column, every straight length (L) is 1 m, so calculation is worked out based on straight length. Inner diameter (D) of adsorber column is 0.04922 m. Therefore, L/D = 1 / 0.04922 = 20.3 which is well above 1, as required by Haselden.

So, total length of adsorber columns

= 5 columns x 2 m

= 10 m

Volume of each adsorber column of length 2 m

= $\pi/4$ x d_i^2 x Column length

= $\pi/4$ x 0.04922^2 x 2

= 0.003805 m^3

Charcoal content of each column = volume × density

= 0.003805 x 550 = 2.093 kg

Therefore, total amount of charcoal accommodated in 5 adsorber columns

= 2.093 x 5

= 10.5 kg

4.3.4 Pressure drop calculation

Pressure drop determination at specified flow rate of 21Nm^3/hr along five activated charcoal columns is shown here in two ways, first one is based on the Ergun equation and other calculation is based on the experimental value obtained from one Indian activated charcoal manufacturer whose sample is analogous to ours.

Ergun equation serves as a model for prediction of pressure drop within packed bed containing particles. The Ergun equation generally used for packed bed calculation is as follows:

Friction factor for packed bed,

$F_p = 150/R_e + 1.75$

$F_p = (\Delta p/L)(D_p/\rho V_s^2)\{Є^3/(1- Є)\}$

Where,

Reynolds Number, $R_e = (D_p V_s \rho) / \{(1- Є)\mu\}$

Equivalent spherical diameter of particle,

D_p= (6 x Volume of the particle) / Surface area of the particle

V_s= Superficial velocity (Volumetric flow rate/Cross sectional area of bed)

Operating pressure of helium gas = 150 bar

Operating Temperature =77 K,

Density,ρ = 70 kg/m^3,C_p=5.38 KJ/kg-K, μ=67.63x10^{-6} Pa-sec,

Volume flow rate = 21 Nm^3/hr,

Particle diameter of charcoal = 3mm (average), Bulk density= 0.55 gm/cc,

Adsorber bed dimensions: OD=60.30mm, ID=49.22mm

Flow rate of helium at 150 bar (a) and 77 k

$= 0.034\ m^3/hr$

$= 9.44\times10^{6}\ m^3/s$

$V_s = 9.44\times10^{-6} / \{ (\pi/4)\ x(0.049222)^2\}$

$= 4.96\times10^{-3} m/s$

$Re = (0.003 \times 4.96 \times 10^{-3} \times 70) / [(1 - 0.6)(51.5 \times 10^{-6})]$

$= 50.6$

So, $F_p = (150 / 50.6) + 1.75$

$= 4.7$

Therefore,

$F_p = 4.7$

$= (\Delta p/10)\ [0.003 / \{70 \times (4.96 \times 10^{-3})^2\}]\ [(0.6)^3 / (1 - 0.6)]$

$\Delta p = 50$ Pa

$= 50 \times 10^{-5}$ bar

It is evident from Ergun equation that the pressure drop within 5 adsorber columns is 50 x 10^{-5} bar, which is not significant in comparison with system pressure.

Table 4.2: Designed values of parameters of adsorber columns in a nutshell

Parameters	**Designed Values**
Adsorber Column Cross section	50 NB Sch 80 SS pipe (OD = 60.30 mm, t = 5.54 mm)
Length of each Adsorber Column	2m,U - shaped with each leg length of 1m
Number of Adsorber Columns	5
Quantity of activated charcoal used	10.50 kg
Theoretical pressure drop in adsorber columns	50 x 10^{-5} bar

4.4 Design of Moisture Separator Vessel

For designing Moisture Separator Vessel, let us consider input impure helium is saturated with moisture at 300K (27^{o}C). The flow rate of impure helium being 20 Nm^3/hr, partial pressure of saturated moisture is 35.70 mbar and its specific volume is 38.78 m^3/kg (data from thermodynamic steam table).

Moisture content of impure helium is (20 / 38.78) kg/hr = 0.51 kg/hr.

After compressing to 150 bar(g) at 300 K, helium volume becomes (20 / 150) m^3/hr = 0.13 m^3/hr and helium is saturated with moisture, and moisture content is 3.35 x 10^{-3} kg/hr. Moisture condensed /hour in Vessel is (0.51 – 3.35 x 10^{-3}) kg/hr = 0.507 kg/hr or water volume is 5.07 m^3/hr.

Let 50NB Sch 80 seamless pipe is selected, let length of pipe required for liquid air collected for 30 minutes be L m.

Therefore, $(\pi/4) \times .04922^2 \times L = 5.07 \times 10^{-4} / 2$

Or, L = 0.133 cm

For cyclonic movement of inlet gas, minimum length requirement is

2 x 0.04922m = 0.09844 m [22].

Minimum length required below inlet is (0.133 +0.09844) m = 0.231 m

Considering the welding of top and bottom cap (0.038 m), drilling of tangential inlet and extra space requirement, fabricated external length of Moisture Separator Vessel comes

out to be 0.3 m. During operation, condensate should be blown down at an interval of 30 minutes.

4.5 Design of Liquid Air Separator Vessel

This separator is cyclone separator. For designing purpose, let us consider 40% nitrogen impurity instead of air impurity in impure helium flowing at the rate of 20 nm^3/hr and delivery pressure 120 bar. Amount of nitrogen remaining after liquid air separator vessel is only 0.83%. Hence, amount of nitrogen separated in liquid air separator is (40 - 0.83)% = 39.17%

which is equivalent to 20 x 39.17/100 = 7.834 nm^3/hr of nitrogen gas.

This amount of nitrogen will be separated as LN_2 which is equivalent to $2.77\times10^{-6} m^3$/sec

Determining the dimension of vessel

Let's calculate with 100NB Sch 80 pipe (OD = 114.30 mm, thickness = 8.56 mm)

Volume of LN_2 produced/15 min= 2.50×10^{-3} m^3

Therefore,

2.50×10^6 $mm^3 = (\pi/4) \times [114.3 - (2 \times 8.56)]^2$

Or, h = 337.05 mm = 0.337 m

Length for cyclonic movement = 2 x inner diameter, [22]

= 2 x (114.30 – 2 x 8.56)

= 194.36 mm

= 0.194 m

Minimum length of vessel = 0.337 + 0.194 = 0.531 m

Adding to it the height of end caps, total height comes out to be 0.580 m.

For the design of the cyclone separator for gas liquid separation, the estimation of settling velocity can be done by the following formula [38],

$u_t = 0.07 [(\rho_L - \rho_v) / \rho_v)^{1/2}$

Where,

u_t = Settling velocity, m/s

ρ_L = liquid density, kg/m^3

= LN_2 density i.e. 813.89 kg/m^3

ρ_v = vapour density, kg/m^3

= Density of helium at 120 bar(g) and 77K = 58.424 kg/m^3

Minimum allowable vessel diameter,[23] $D_v = [(4V_v) / (\pi \times 0.15 \times u_t)]^{1/2}$

From the above equation we get, $D_y = 0.02$ m

Hence, our selection of 100NB Sch 80 pipe is suitable.

Maximum blow down interval can be calculated when 40% nitrogen impurity flowing at rate of 20 nm^3/hr at 150 bar with helium.

Rate of LN_2 accumulation in vessel = 3.09×10^{-6} m^3/s

Max.blow down interval = LN_2 accumulation in 15 min/LN_2 formation rate

= $[2.78\times10^{-3}/3.09\times10^{-6}/60]$ min

= 15 min

4.6 Design of Tube-in-Tube Heat Exchanger

A typical double pipe heat exchanger consists of one pipe placed concentrically inside another of larger diameter with appropriate fitting to direct the flow from one section to another next. The major use of double-pipe heat exchanger is for sensible heating or cooling of process fluids, where small heat transfer area is required.

The tube-in-tube heat exchanger is made by 6 m length of 2 sections and welded to get 12m length of seamless tube of SS 316L. Tube-in-tube heat exchanger was designed to cool impure helium from 300 K to 120 K and to heat pure helium from 77 K to 300K.Outer tube dimensions through which impure helium flows are 10 mm (OD), thickness 1mm and inner tube dimensions through which pure helium flows are 16 mm (OD), thickness 1.5 mm. Practically we got the impure helium flows in outer tube which cool down from 274 K to 120 K and pure helium flows in inner tube which is heated from 77 K to 270 K. The calculations are as follows:

Impure helium is cooled from 274 K to 120 K, T_{h1}=274 K, T_{h2}=120 K

Pure helium is heated from 77 K to 270 K, T_{c1}=77 K, T_{c2} = 270 K

Mass flow rate of helium in outer tube is (m_{hHe}) 38 l/min = 0.47 kg/sec

Mas flow rate of nitrogen in outer tuber is (m_{hN2}) 2 l/min = 0.02 kg/sec

Specific heat of helium at 274 K and pressure of 120 bar $(C_{pHe})_h$ = 5.1884 kj/kg k

Specific heat of nitrogen at 274 K and pressure of 120 bar $(C_{pN2})_h$= 1.2732 kj/kg k

Specific heat of helium at 77 K and pressure of 120 bar $(C_{pHe})_c$ = 5.4125 kj/kg k

We can calculate mass flow rate of pure helium (m_{cHe}) coming at 77 K from adsorber columns.

From heat balance:

$Q_h = Q_c$

$[m_{He}\ C_{pHe} + m_{N2}\ C_{pN2}]_h \Delta T_h = (m_{He} C_{pHe})_c \Delta T_c$

$[(0.47\times5.1884) + (0.02\times1.2732)]\ (273-120) = [(m_{cHe}\times5.4125)]\ (270-77)$

Therefore,

$m_{cHe} = 0.3628$ kg/sec

The capacity rates may be determined:

$C_h = [m_{He}\ C_{pHe} + m_{N2}\ C_{pN2}]_h = [(0.47\times5.1884) + (0.02\times1.2732)]$

$C_h = 2.46 = C_{max}$

$C_c = (m_{He} C_{pHe})_c = (0.3628\times5.4125)$

$C_c = 1.96 = C_{min}$

The cold fluid is the minimum-capacity fluid; therefore,

$C_R = C_{min}/C_{max} = 1.96/2.46 = 0.797$

The effectiveness may be determined directly:

$$\varepsilon = \frac{T_{c2}-T_{c1}}{T_{h1}-T_{c1}}$$

$$\varepsilon = \frac{270-77}{274-77} = 0.979$$

The NTU is,

$$\text{NTU} = \frac{1}{1-C_R} ln\left(\frac{1-C_R\varepsilon}{1-\varepsilon}\right)$$

$$\text{NTU} = \frac{1}{1-.797} ln\left(\frac{1-(0.797)(0.979)}{1-0.979}\right)$$

NTU = 11.60

For Over-all heat transfer (U):

$NTU = UA/C_{min}$

Where, Area of heat transfer (A) $= 2\pi d_o L$

$= 2\times\pi\times0.01\times12$

$= 0.745\ m^2$

$11.60 = U\times0.754/1.9636$

$U = 30.20\ kw/m^2k$

Therefore our designed tube in tube heat exchanger is suitable for purifier system to condense air impurities at 120 bar and 77 K.

4.7 Analysis of LN_2 vessel design and LN_2 Consumption

LN_2 vessel in which all the major components of purifier are housed inside and arranged in such a manner to optimize the size of LN_2 vessel as inside diameter and depth of inner vessel comes out to be 600 mm and 1600 mm respectively. It is designed as per ASME Section VIII Division I and made up of SA 240 TP 304 L

.

4.7.1 LN_2 Consumption

LN_2 vessel is partially filled with LN_2 for maintaining low temperature at various section of purifier. At the bottom of the LN_2 vessel, adsorber columns are situated above which liquid-air separator vessel is placed and then two heat exchangers. So the adsorber column and liquid-air separator vessel is completely submerged in LN_2 for efficient adsorption of impurities and removal of condensate air. Subcooler heat exchanger is also completely submerged in LN_2 for removing air impurities through proper condensation and tube-in- tube heat exchanger is in the vapour region LN_2 vessel.

During Operation LN_2 consumption can be evaluate by considering static evaporation loss, LN_2 consumed due to adsorption heat evolution and LN_2 boil-off for nitrogen impurity condensation.

The maximum and minimum LN_2 level in LN_2 vessel during purification operation is designed to be 1410 mm and 1210 mm respectively.

The basic input parameters are as follows:

Inside diameter of inner vessel of LN_2 vessel = 592 mm

Outside diameter of inner vessel of LN_2 vessel = 600 mm

Inner vessel height = 1600 mm

Ambient temperature, T_2 = 300 K

Number of layers of superinsulation = 20 layers

Latent heat of LN_2, h_{fg},= 199.30 kJ/kg

Density of LN_2 at 77 K = 813.89 kg/m^3

C_p of helium = 5.20 kJ/kg-K

C_p of N_2 = 1.142 kJ/kg-K

Density of helium at 300 K = 0.16 kg/m^3

Density of nitrogen at 300 K = 1.124 kg/m^3

Emissivity of highly polished top flange surface, ε = 0.028 [1]

Thermal conductivity of SS 304 at 300 K, k_{ss} = 14.90 W/m-K [2]

Capacity of empty LN_2 vessel

$= \{(\pi/4) \times 0.592^2 \times 1.41\}\ m^3$

$= 388$ lit.

1350 mm is average LN_2 level in LN_2 vessel when in operational condition and by taking the average LN_2 level calculation has been done. Average LN_2 level is 0.25 m from the bottom surface of the top flange.

Heat transfer by radiation through shell and dish end is as follows:

Radiation heat transfer [1] through shell and dish end of LN_2 vessel having superinsulation as radiation shields,

$$Q_1/A = \sigma \times F_e \times F_{1\text{-}2} \times (T_2^4 - T_1^4)$$

Where, Stefan Boltzmann Constant, $\sigma = 5.67 \times 10^{-8}\ W / m^2 K^4$

Configuration factor, $F_{1\text{-}2} = 1$, as inner vessel is completely enclosed by outer vessel Emissivity factor for 20 layers of superinsulation,

$$F_e = [2 \times (1/e_0 + 1/e_s - 1) + (n_s - 1)(2 - e_s) / e_s]^{-1}$$

Where,

e_0, emissivity of inner and outer surface of vessel which is polished surface of stainless steel = 0.028 [1]

e_s, Emissivity of superinsulation = 0.04 [1]

n_s, Number of layers of superinsulation = 20

Thus, $F_e = [2 \times (1/0.028 + 1/0.04 - 1) + (20 - 1)(2 - 0.04) / 0.04]^{-1}$

$= 9.52 \times 10^{-4}$

$Q_1/A = 5.67 \times 10^{-8} \times 9.52 \times 10^{-4} \times 1 \times (300^4 - 77^4)$

$= 0.435\ W/m^2$

Inside surface area of shell and dish end of inner vessel

$= [(\pi \times 0.592 \times 1.556) + 0.303]\ m^2$

$= 3.197\ m^2$

Q_1, Radiation heat transfer

$= (0.435 \times 3.197)\ W$

$= 1.39\ W$

Q_2, Heat radiated from top flange

$= \sigma A \varepsilon T^4$

$= (5.67 \times 10^{-8}) \times \{(\pi/4) \times 0.592^2)\} \times 0.028 \times (300)^4$

$= 3.54\ W$

Q_3, Heat conducted through shell wall surface from top end

$= k_{ss} A \, dT/dx$

$= 14.90 \times (\pi \times 0.592 \times 0.004) \times (300 - 77) / 0.25$

$= 98.87$ W

Heat flux conducted, Q_4, through tubes of sizes (OD 6 mm x 1 mm, OD 10 mm x 1 mm) at ports P1, P2, P3, P4, P5, P6, P10 [refer Fig. 3.1 for nomenclature] of the top flange is shown here under:

Cross section area of 3 tubes (OD 6 x 1) through P2, P3, P5

$= \pi \times 0.006 \times 0.001 \times 3$

$= 5.7 \times 10^{-5}$ m^2

Cross section area of 4 tubes (OD 10 x 1) through P1, P4, P6, P10

$= \pi \times 0.01 \times 0.001 \times 4$

$= 1.3 \times 10^{-4}$ m^2

Total cross section area, A

$= (5.7 \times 10^{-5} + 1.3 \times 10^{-4})$ m^2

$= 1.87 \times 10^{-4}$ m^2

Q_4, Heat conducted through the tubes connected to top flange

$= k_{ss} A \, (dt/dx)$

$= 14.90 \times 1.87 \times 10^{-4} \times (300 - 77) / 0.25$

$= 2.5$ W

Total Heat flux input

$= Q_1 + Q_2 + Q_3 + Q_4$

$= (1.39 + 3.54 + 98.87 + 2.5)$ W

$= 106.30$ W

$= 9.18 \times 10^6$ J/day

$= 56.59$ l of LN_2 evaporation / 24 hr

$= 14.13$ litre of LN_2 evaporation / 6 hr

Therefore, static LN_2 evaporation loss for 6 hr is 14.13 litre.

4.8 Selection of Regeneration Process

There are two types of regeneration process:

1) The Pressure Swing Cycle
2) The Thermal Swing Cycle.

In pressure swing regeneration technique, the saturated adsorbent columns are purged with low pressure, non-adsorbing, pure gas isothermally. In thermal swing cycle, the adsorbent columns are heated to proper reactivation temperature of that adsorbent material and desorbed impurities are flushed out by non-adsorbing gas or by vacuum pump. Thermal swing process does not have deleterious effect on adsorbent, if excessive temperature is not used.

Two methods of thermal swing regeneration technique are there, one is heating and simultaneous back flow of Grade 4.5 helium gas through adsorber columns, and the other is heating and evacuation. We have opted for heating and evacuation regeneration technique for the helium purifier as this process does not need Grade 4.5 helium. In this process, activated charcoal is heated to 120^{o}C for desorption of gases for 4 hr, and is evacuated with rotary pump to get 10^{-3} mbar pressure. Then system is back filled with Grade 4.5 helium. The purity of back filled helium is monitored with Linde multi-component detector at purifier inlet for ensuring proper regeneration.

Tubular Heaters

The adsorber columns are heated by six tubular heaters positioned between adsorber columns within the LN_2 vessel. The heater are chosen according to specification 1 kW, 220 VAC, single phase, 50 Hz. Tubular heaters have been arranged in inside LN_2 that uniform heating of adsorber columns takes place.

Time Estimation for Warm Up

During regeneration of purifier, the components which are warmed up by heaters are tube-in-tube heat exchanger, subcooler, liquid air separator vessel, and snow filter, adsorber columns, inner vessel of LN_2 vessel, middle portion of top flange exposed to radiation heat, cage, connecting tubes and fittings. Heaters raise the temperature of these components from 77 K to 393 K.

The input data for estimation are the following:

Mass of activated charcoal in adsorber columns, $m_{charcoal}$ = 10.5 kg

Mass of adsorber columns without activated charcoal = 105 kg

Mass of 2 heat exchangers, liquid air separator vessel and snow filter = 28 kg

Mass of inner vessel of LN_2 vessel and middle part of the top flange exposed to radiation heat = 159 kg

Mass of cage, tubing and fittings = 26 kg

m_{SS}, Total stainless steel mass heated by tubular heaters

= (105 + 28 + 159 + 26) kg

= 318 kg

$(C_p)_{charcoal}$, Specific heat capacity of wood charcoal = 1 kJ/kg.K [40]

$(C_p)_{SS}$, Specific heat capacity of stainless steel = 0.46 kJ/kg.K [40]

Total heat required to raise temperature of purifier to 393 K.

$= m_{charcoal} \times (C_p)_{charcoal} \times 316 + m_{SS} \times (C_p)_{SS} \times 316$

= (10.5 x 1 x 316 + 318 x 0.46 x 316) kJ

= 49542.5 kJ

Power of 6 heaters = 6 kW

Time required

= (49542.5 / 6) s

= 8257 s

= 2.29 hr

Minimum time required to heat up the purifier is 2.29 hr assuming no heat loss. Hence, we have chosen regeneration time of 4 hours, during which heating and evacuation go on simultaneously. Fig. 3.8 shows schematic diagram of a tubular heater.

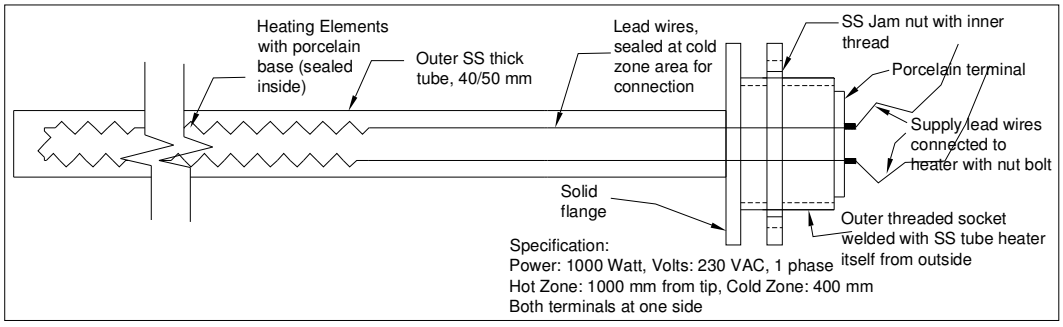

Fig. 4.6: Schematic diagram of a tubular heater

CHAPTER 5

DESIGN AND DEVELOPMENT OF HELIUM PURIFIER

CHAPTER 5

DESIGN AND DEVELOPMENT OF HELIUM PURIFIER

The helium purifier is a compact and reasonably complex system. All the cryogenic components of the system are housed within a Superinsulated LN_2 vessel. The purifier is made compact in order to reduce LN_2 consumption for cool down. Helium purifier works in the temperature range of -196^oC to 120^oC and endures a large range of pressure viz. 150 bar to 10^{-3} mbar. The demountable joints and valves have helium leak tightness of 10^{-6} mbar-l/s or better. The joints which are exposed to LN_2 temperature and high pressure were TIG welded and radiographed. The development of helium purifier involved the selection and fabrication of high pressure machinery, high pressure storage and vacuum system, cryogenic system and its fool proof safety devices, piping network interconnecting all components, and a centralized control panel from where a single operator can operate the total system.

The list of machine tools used for constructing the purifier system is as follows: TIG welding machine, electric arc welding machine, plasma cutting machine, lathe, grinder cutter, drill, buffing tool, Swagelok tube bender, deburring tool and cutter, radiograph machine.

Items for fabrication of the components were procured from several vendors in India and abroad. All welding were performed by welder who was qualified to procedures and positions as required by ASME Section IX, Article II, of the Boiler and Pressure Vessel Code. All tube/pipe welding at site met the requirements of ASME B31.3 Piping Code, Chapter V for full penetration welds.

5.1 Fabrication of major Components of Helium Purifier system

Major components of purifier are:

i) Moisture collector and Liquid Air separator vessels
ii) Shell and Tube Heat Exchanger
iii) Tube in Tube Heat Exchanger
iv) Helical Coiled Tube Heat Exchanger
v) Snow Filter

vi) Adsorber Columns

vii) Cylinder Manifold

5.2 Fabrication of Moisture Collector and Liquid Air Separator Vessels

Shape, fabrication procedure and testing of both moisture collector vessel and liquid air separator vessel are same, only the vessel diameters and lengths are different. The materials used for the construction of both the vessels are the following:

(1) For moisture collector vessel, 50 NB Sch 80, seamless, ASTM A312 TP 316L

(2) Pipe for liquid air separator vessel, 100 NB Sch 80, seamless, ASTM A312 TP 316L

(3) End caps are, 50 NB Sch 80 and 100 NB Sch

(4) Tube, 6 mm OD, thickness 1 mm, SS 316L

(5) Tube, 10 mm OD, thickness 1 mm, SS 316L

The vessels were fabricated by welding two end caps at both ends of SS pipe. Length of moisture collector vessel and liquid air separator vessel are 370 mm and 640 mm respectively. The pipes were drilled at an angle such that 6 mm OD and 10 mm OD tubes enter tangentially to the inner surface of pipes of moisture collector vessel and liquid air separator vessel respectively..

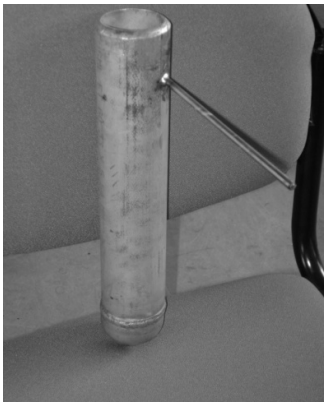

Fig. 5.1: Liquid air separator vessel under fabrication

5.3 Fabrication of Shell and Tube Heat Exchanger

Shell and tube heat exchanger, located after the moisture collector vessel, eliminates the remaining moisture by bringing down the water dew point of impure helium gas to 1^{o}C by the cold purified helium. In this counter-flow heat exchanger, the impure helium flows through the shell, while the cold helium gas runs through the coil.

The materials used for the construction of shell and tube heat exchanger are the following:

(1) Pipe, seamless, size 50 NB Sch 80, ASTM A312 TP 316L

(2) End cap, seamless, 50 NB Sch 80, forged, seamless with butt weld ends

(3) Tube, 6 mm OD, thickness 1 mm, SS 316L

(4) Socket fittings for the tubes

The shell was made by welding two end caps with pipe. Coil was fabricated out of 6 mm OD tube. Fig. 4.2 shows the bottom part of the heat exchanger under fabrication.

Fig. 5.2: Shell and tube heat exchanger before closing one end

5.4 Fabrication of Tube-in-Tube Heat Exchanger

Tube-in-tube heat exchanger is a counter-current flow exchanger where warm impure helium flows through outer tube and cold purified helium passes through inner tube. Tube-in-tube heat exchanger [1][2] consists of two concentric tubes coiled helically. This design is very compact and robust which prevents thermal fatigue, increases efficiency, reduces overall size, maintenance free, cost effective and is ideal for high pressure and low flow rate application.

Tube-in-tube heat exchanger was fabricated out of 10 mm and 16 mm OD seamless SS 316L tubes of standard length. Length of heat exchanger is 12 m and is all welded construction. As the length of heat exchanger is 12 m, so it was fabricated into two segments of 6 m length each and then welded. Copper wire of 1 mm diameter, acting as spacer, was helically wound over 10 mm OD tube and soldered at terminals as well as at several spots in between. Then this tube was inserted within 16 mm OD tube properly. The use of copper wire spacer allows the gas in the annular space to follow a longer

helical path which results in the increase in fluid velocity and heat transfer coefficient. After fabrication, high pressure nitrogen was purged from both ends of the coil to clean the annular space and inner tube. Fig.4.3 shows the tube-in-tube heat exchanger after fabrication.

Fig.5.3: Tube-in-tube heat exchanger\

5.5 Fabrication of Subcooler Heat Exchanger

Subcooler heat exchanger condenses the air contaminants in impure helium stream at 77 K and reduces the impurity load of the helium entering the adsorber columns. This is a single helical coiled tube designed for purifier delivery pressure of 150 bar.

Subcooler was fabricated out of 2 tubes of 6 m length each i.e. total length is 12 m. Tube size is 6 mm OD x 1 mm thickness and material of construction is SS 316L. The coil diameter was made 490 mm to fit the cage dimensions.

5.6 Fabrication of Snow Filter

Snow filter functions as eliminator of ice crystals of carbon dioxide and water particles. It was made by welding two end caps (50 NB Sch 80, seamless with butt weld ends, ASTM A312 TP316L) at both the ends of 50 NB Sch 80, SS 316L pipe. Length of the snow filter is 120 mm. The inlet gas port is at top and outlet port is located horizontally at the centre. Four layers of 40 mesh SS wire cloth roll was inserted which acts as filter. Fig. 4.4 shows the snow filter before welding one end cap. Inside wire cloth and two ports are seen in photograph.

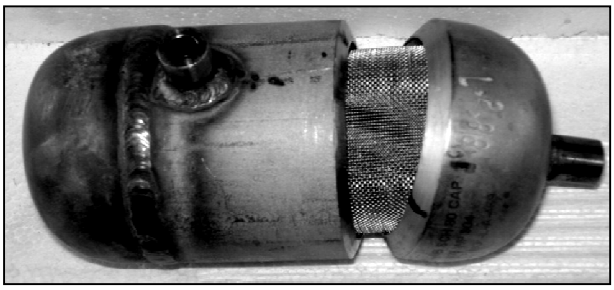

Fig. 5.4: Snow filter before welding

5.7 Fabrication of Adsorber Columns

Adsorber columns are 5 in number and each column is U-shaped having three parts: two straight lengths, top cap at both ends and a U-bend at the bottom. The materials used for the construction of adsorber columns are the following:

(1) Pipe, seamless, size 50 NB Sch 80, ASTM A312 TP 316L

(2) U-bend, long radius forged, 50 NB Sch 80, seamless with butt weld ends

(3) End cap, 50 NB Sch 80, forged, seamless with butt weld ends

(4) Wire mesh, 40 mesh, wire diameter 0.224 mm, SS 316L

(5) Perforated plate for filter, 3 mm sheet thickness, SS 316L

(6) Round bar for filter housing, 50 mm diameter, 20 mm length, SS 316L

(7) Round bar for plug of adsorber column, 30 mm diameter, SS 316L

(8) Round bar for top end socket, 12 mm diameter, SS 316L

(9) Activated charcoal for adsorption

(10) Molecular sieve to act as filter to charcoal dust in addition to adsorbing gases

Column

The fabrication procedure explained here is for one adsorber column and same for all columns. Two pieces of 1 m length pipe were cut and edges were prepared by grinding. Edges of end caps and U-bends were prepared in a similar manner. At the bottom of U-bend, a plug and a socket were welded for possible charcoal replacement. For replacing charcoal in future, one can remove the plug by cutting the weld with a grinder cutter. After refilling, the same plug can be rewelded with socket. Fig. 4.9 depicts the dimensions of plug and socket. The U-bend was welded to the pipe pieces after making proper alignment. And sockets were welded with the end caps of adsorber column. The top end socket diagram is shown in Fig. 5.7.,Fig.5.6 shows the longitudinal sectional view of the adsorber column.

Filter Unit

The filter unit consists of one perforated plate and four layers of wire mesh which were placed within the filter housing. Filter housing was machined out of 50 mm round bar, as shown in Fig. 5.8. Top layer is perforated plate and four layers of wire mesh are below it. Holes in perforated plate are of 5 mm diameter with 3 mm spacing in between them in staggered way. The perforated plate was tack welded to filter housing at three points.

Fig. 5.5: Filter unit of adsorber column

Packing of Activated Charcoal

After welding of U-bend to pipe pieces, activated charcoal was tightly packed in the pipes. For packing, some quantity of charcoal was poured into the column, which was tapped with wooden mallet on the outer surface continuously and compressed with a wooden piece from the top. This process was continued till the column was full. Compaction is a tedious and time consuming process. Molecular sieve was packed at the top of activated charcoal to make a layer of 75 mm thick at both ends of adsorber column.

Passivation of Welds

After radiography, the Heat Affected Zone (HAZ) was passivated with 20% (by volume) nitric acid solution for around one hour at room temperature. Before passivation, the welded spots were cleaned with SS wire brush to remove loose surface oxidized contaminants, and then degreased with acetone.

Assembly of adsorber columns along with heat exchangers is shown in Fig. 5.6.

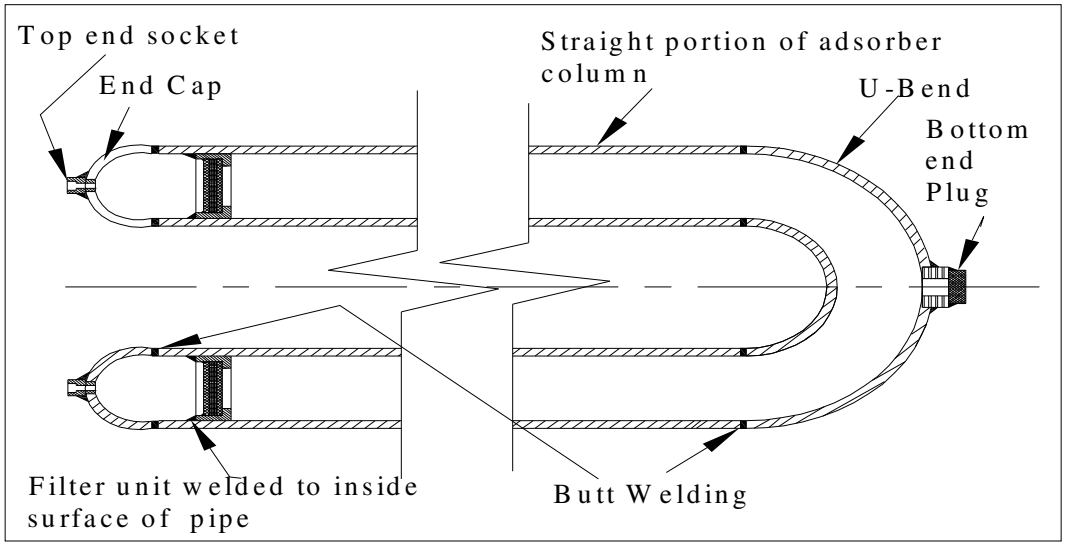

Fig. 5.6: Sectional view of the adsorber column

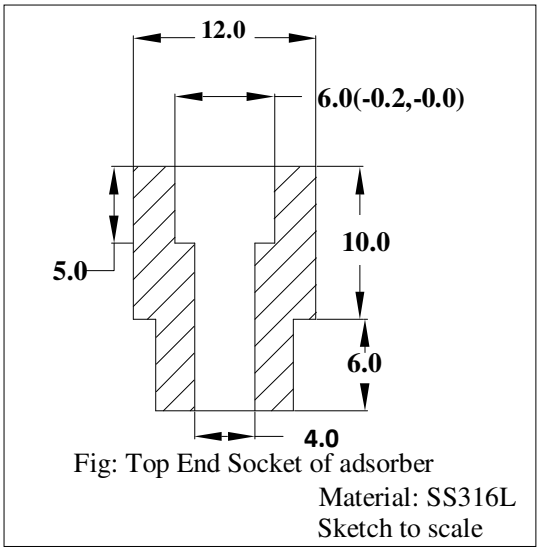

Fig. 5.7: Top end socket of adsorber column

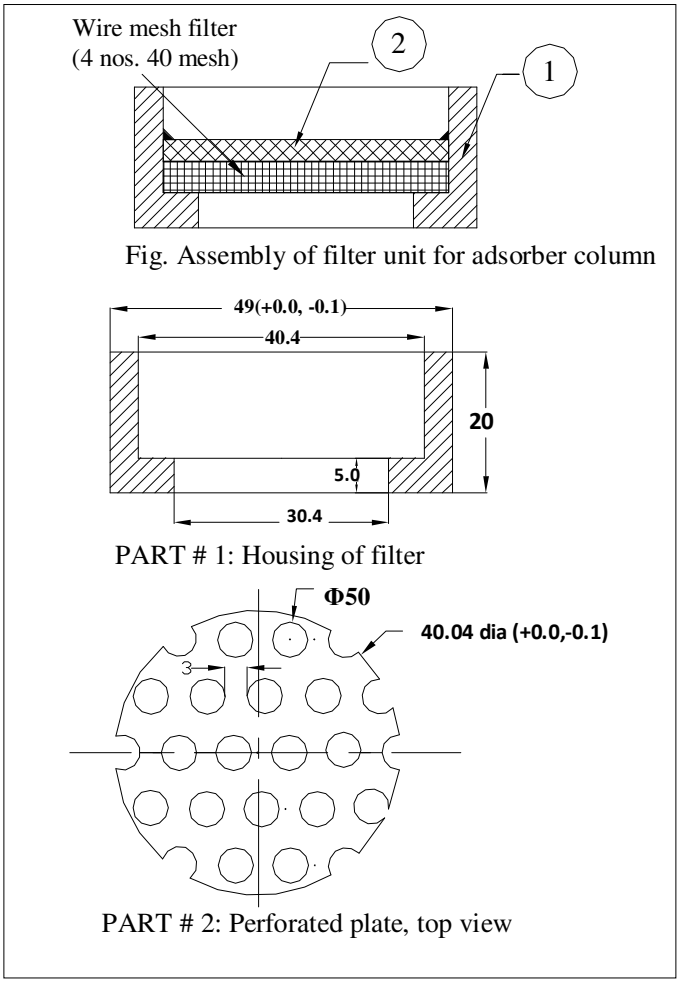

Fig. 5.8: Components and assembly of filter unit

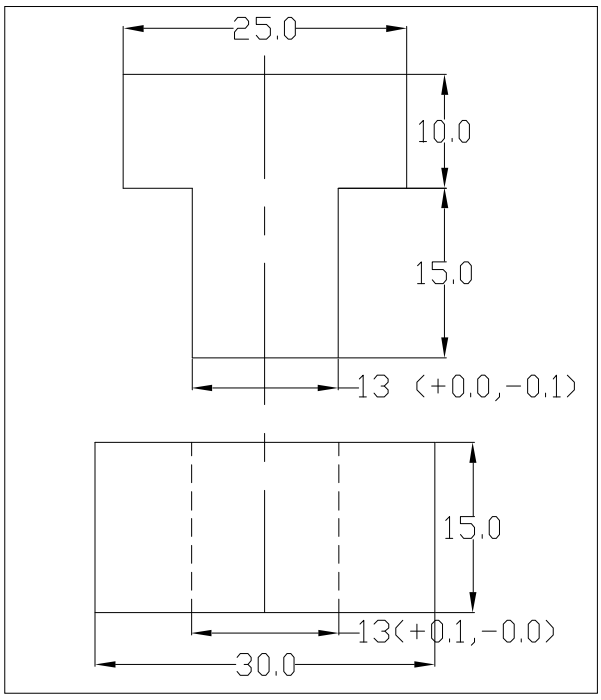

Fig. 5.9: Bottom end plug and socket of adsorber column

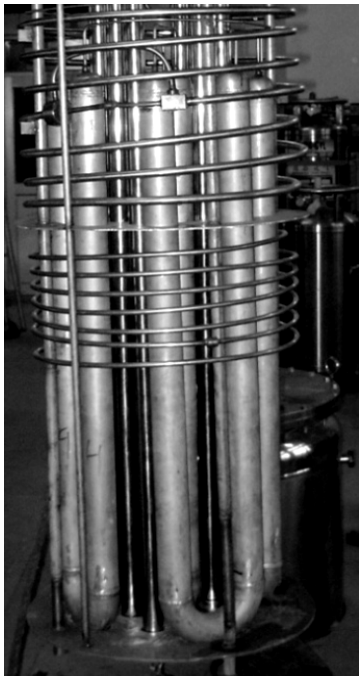

Fig. 5.10: Adsorber columns in assembled condition

5.8 Fabrication of High Pressure Cylinder Manifold

The system contains two cylinder manifolds: the first one is a high pressure gas storage system which supplies impure helium to purifier for purification and the second one receives Grade 4.5 helium from purifier at 150 bar. Each manifold consists of 10 cylinders. The cylinder manifold assembly consists of a pressure reducing regulator, change over devices, shut-off valves, gauges, pressure relief valve and so on. All the tubes are of copper as it is cheaper than stainless steel with respect to material cost as well as fabrication cost. Working pressure of each manifold is 150 bar at ambient temperature. Cylinders used in this experiment are complying IS: 7285 and having water capacity of 47 l. Pig tail tube material is copper and nipple is of brass. Cylinder valves are oxygen valves complying IS:3224 and of spindle type and Indian manufacture. Header tubes are of size ¾" Outer Diameter x ½" Inner Diameter and pig tail of size 3/8" OD x 16 SWG. The tubes are high pressure copper tubes and complying ASTM B42 C12200. Due to high pressure application, brass blocks and nipples were machined from high tensile brass rods conforming to BS: 2874/86 CZ 115 and are equivalent to BS 1001.

A needle valve was placed at the end of each cylinder manifold for evacuation and purging during commissioning and refurbishing of manifolds.

Fig.5.11: Cylinder Manifold

5.9 Fabrication of LN_2 Vessel

LN_2 vessel is double walled superinsulated vessel which consists of two parts:

(1) Top flange

(2) Vessel, which is superinsulated

Fig. 5.12 illustrates the location of ports on the top flange. LN_2 is filled through port, P4 and boil off vents through port, P7. All the ports are arranged along the circle having pitch diameter 500 mm. The right hand side ports are for passing low temperature gas and LN_2, and left hand side ports are for ambient temperature gases.

Table 5.1: Specification of LN_2 vessel

Sl No.	Parameter	Value
1	<u>Inner Vessel</u> Outside diameter, Depth Shell thickness, Dished end thickness Material of construction	 600 mm, 1600 mm 4 mm, 5 mm SA 240 TP 304 L
2	<u>Outer Vessel</u> Outside diameter Shell thickness, Dished end thickness Material of construction	 700 mm 4 mm, 5 mm SA 240 TP 304 L
3	Top and Bottom flange thickness (Bottom flange has neoprene O-ring)	22 mm
4	Superinsulation material Make	20 layers of doubly aluminized Mylar with nylon netting spacers, 10 layer blanket Austrian Aerospace Co.
5	<u>Annular Space</u> Gap, Vacuum Adsorbent used	 46 mm, 10^{-6} mbar Charcoal
6	Helium leak tightness of welds	1 x 10^{-9} mbar-l/s
7	Evacuation port size	ISO KF 25

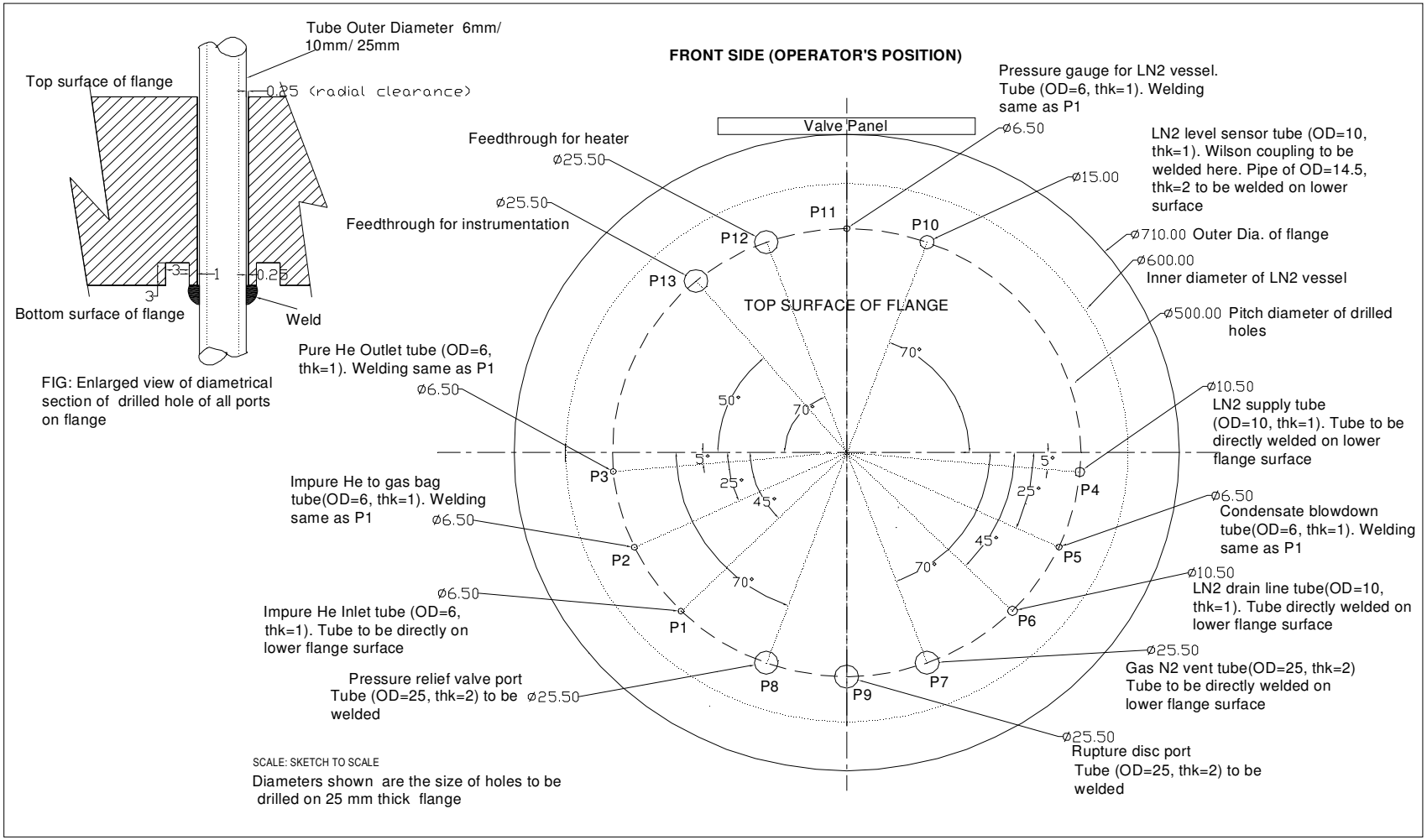

Fig. 5.12: Location of ports on the top flange of LN_2 vessel

5.10 Assembly of the Helium Purifier

The outside height of the LN_2 vessel of purifier is 1700 mm. As the operator operates standing in front of valve panel, LN_2 vessel has been placed in a pit within the laboratory. For material handling, movable gantry crane was used.

Before the beginning of assembly process, the components were given a final and thorough checking. Superinsulated LN_2 vessel was designed in such a way that all the components were accommodated in that space in a very compact manner for minimum consumption of LN_2.

Fabrication of Cage

All the internal components of purifier unit were arranged within a cage which was suspended from the top flange of the LN_2 vessel. This cage consists of three circular plates of 562 mm diameter and 3 mm thickness, and 4 tie rods. Tie rods are 15 NB ASTM A312 TP316L pipe of length 1482 mm.

Two of the three plates, viz. Top and middle ones were tack welded to the tie rods, and the bottom plate was fully welded with the tie rods. The top plate of cage is located 110 mm below top flange. The openings were made in all three plates by plasma cutting machine. The top plate has six openings to support six tubular heaters.

The middle plate, located 510 mm below the top plate, positions adsorber columns and heaters such that there is sufficient gap between them. Bottom plate, located 802 mm below the middle plate, supports the adsorber columns.

Assembly of Components

All the adsorber columns were placed in cage after welding of middle and bottom plate with tie rods. The columns were connected by 6 mm OD tubes for flexibility during thermal expansion and contraction. We fixed tube-in-tube heat exchanger, subcooler, liquid air separator vessel and snow filter, with the cage and interconnected them with tubes and fittings. Subcooler, liquid air separator vessel and snow filter were placed between the middle and the bottom plate and were tack welded to tie rods by using clits. Subcooler was wrapped around the tie rods and clamped with it. Tube-in-tube heat exchanger was placed between the top and the middle plate and was wrapped around tie rods and clamped with it. After fastening heaters with the top plate, it was tack welded with the tie rods. The top plate was placed such that it is above maximum LN_2 level of the vessel.

Tie rods were welded to bottom surface of the top flange through short pieces so that the cage can be detached from flange for modification and maintenance purpose by grinding the short pieces weldments. The tubes from internal components were welded to port P1, P2, P3 and P5 [refer Fig. 3.1 for nomenclature] on the top flange.

The assembly of internal components along with the top flange was lifted by movable gantry crane and mounted on the LN_2 vessel. Valve panel was welded on the top flange. All the valves, pressure gauges and back pressure regulator were fixed to valve panel. Back pressure regulator was set at 120 bar(a) by operating compressor. The assembly process was completed by purging the system with high pressure dry nitrogen in order to remove the traces of dusts and particles. Fig.4.12 shows the assembly process.

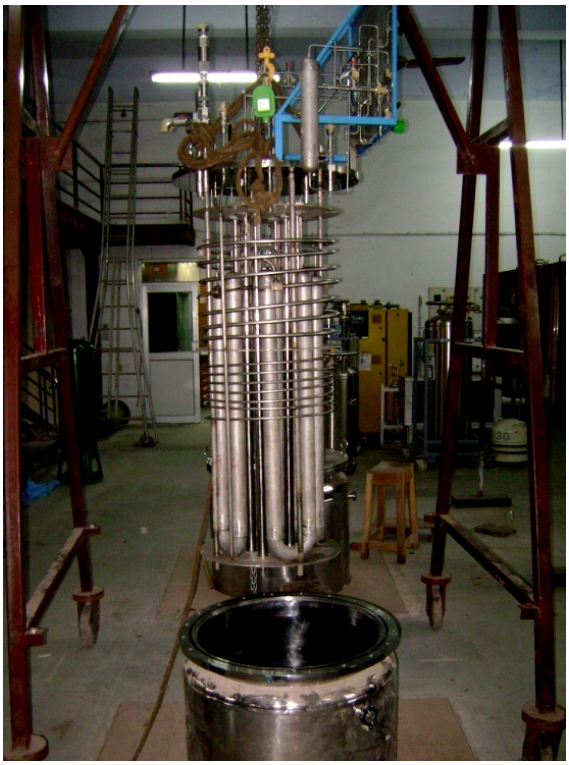

Fig. 5.13: Assembly process of helium purifier

5.11 Inspection and Testing of Helium Purifier

The helium purifier is a high pressure cryogenic device, so non-destructive tests were carried out on the fabricated components according to rules and guidelines prescribed by ASME B 31.3.

After successful completion of the assembly of purifier, the pneumatic leak test was performed with nitrogen gas at test pressure of 165 bar(g) in accordance with ASME B 31.3. Test pressure was retained for 10 minutes and pressure drop was observed. No pressure drop was found. Then system pressure was reduced to 150 bar(a) i.e. design pressure. At this pressure all the joints which include welds, threaded joints, ferrule fittings were checked by soap solution as well as pressure drop was observed in gauge. This pressure was held for 30 minutes for total checking. No leakage was witnessed.

CHAPTER 6

EXPERIMENTAL PROCEDURE AND RESULTS

Chapter 6

EXPERIMENTAL PROCEDURE AND RESULTS

This chapter illustrates about the experimental set up, procedure and testing of helium purifier. Helium purifier works on cryocondensation and cryoadsorbtion process, so before going to perform operation of purification commissioning and conditioning should be done.

6.1 Conditioning of helium purifier

After installation of all components the purifier should be evacuate by using rotary vacuum pump up to 10^{-3} mbar for 3 days. While evacuating purifier was heated by tubular heaters for complete removal of moisture from purifier and charcoal adsorber columns. Meanwhile the system was purged with grade 4.5 helium and this process was repeated till the quality of the purged gas coming out from adsorber columns was same as that of the input gas i.e. Grade 4.5. The purifier was pressurized with Grade 4.5 helium and the system was ready for purification process.

6.2 Operation of Purifier system

The operation of the purifier was conducted once for one session and for this experiment grade 4.5 helium and dry nitrogen is used. As two cylinder manifolds were there, one manifold was used to connect 10 helium cylinders which were utilized to prepare impure helium in gas bag. And in the other manifold, 10 empty cylinders were connected for collection of purified helium from the purifier. Pure helium is supplied from cylinder manifold passing through rotameter at the flow rate of 38 l/min and nitrogen gas at the flow rate of 2 l/min to gas bag at 50 mbar. The mixture of pure helium and nitrogen makes impure helium in gas bag i.e. about 5% impurity and 95% pure helium. This impure gas was compressed by compressor to deliver the impure helium at pressure of 150 bar at flow rate of 20 nm^3/hr for purification to purifier cylinder. The purified gas from purifier was collected in empty sample cylinders.

Operation of purifier undergoes the following modes:

(1) Cooling down of purifier

(2) Purification

(3) Regeneration

Cooling down

In purifier system it is necessary to maintain LN_2 temperature, so LN_2 vessel was filled with LN_2 at pressure of 2 mbar by using pressurization system of Dewar. LN_2 level in the vessel was 1300 mm which was measured by stainless steel dip stick and which required 300 litres of LN_2.

Purification

In purification process, the impure helium from gas bag at 50 mbar is supplied to inlet of the compressor. From the outlet of the compressor the impure gas releases at 120 bar and deliver it to purifier at a flow rate of 20 nm^3/hr. The impure helium once passed from Back Pressure Regulator to BV6 valve (refer Fig. 3.1 for nomenclature) which is opened at the time initiation of compressor then BPR should is set at 120 bar. BPR maintains pressure of 120 bar inside the purifier for proper condensation and adsorption. During the operation, valves BV2, BV3, BV5 were purged for few seconds every half an hour in order to prevent overflow of collected condensate. The purifier was operated for one session and was stopped by switching off compressor and closing BV6. The purified gas was collected in empty cylinders in manifold at 120 bar(a).

There are four sample cylinders of Swagelok (water capacity 75 cc) in which purified gas are collected at regular interval of time, which were sent to VECC, Kolkata, for analysis.

Fig.6.1: Picture of running operation of helium purification system

Regeneration

After purification, LN_2 was drained off from LN_2 vessel for another session of purification. Regeneration of the purifier was carried out by heating and evacuation for 2-3 days. The evacuation was done by vacuum rotary pump through BV5, BV1 and grade 4.5 helium was purged time to time for complete removal of moisture from the pipes and charcoal adsorber columns.

6.3 Results of the Experiment

Sample cylinders are filled only after 45 minute running of purifier. First sample cylinder collected purified gas after 45 minutes then other three cylinders were filled at an interval of 30 minute. The purity of output helium was checked off-line by feeding samples from sample cylinders to Linde Multi-Component Detector, at Variable Energy Cyclotron Centre, Kolkata. The brief specification of Linde Multi-Component is as follows:

Make : Linde Kryotechnik AG, Germany
Model : WE37M-3
Measuring range : 1 to 100 vpm (ppm by volume) H_2O in pure helium
: 1 to 100 vpm N_2 in pure helium
: 1 to 60 vpm O_2 in pure helium
Resolving power : 0.10 vpm
Reproducibility : ± 0.10 vpm
Measurement principle: Optical emission spectroscopy
The analysis of the four samples of purified helium is presented in Table 5.1.

Table 6.1: Results of the four samples

Sl No.	Sample No.	H_2O (vpm)	N_2 (vpm)	O_2 (vpm)	Total impurities (vpm)
1	1	1.70	2.10	0.20	4
2	2	2.30	1.50	0.10	3.9
3	3	1.40	1.40	0.30	3.1
4	4	1.50	2.80	0.30	4.6

The results show that in all the samples total impurities, consisting of moisture, nitrogen and oxygen is less than 5 vpm, which means purified helium is better than Grade 4.5 or 99.995% helium.

CHAPTER 7

CONCLUSION

Chapter 7

CONCLUSION

The purifier is designed to purify upto 40% impurity to give 4.5 grade or 99.995% pure helium by high pressure and low temperature cryosorption process. In our first experiment or first purification session we have added 5% impurity of dry nitrogen in 99.995% pure helium and after experiment we got that the 5% impurity reduces to 5 ppm, where in pure helium 5 ppm impurity is allowable.

Summarizing, the following may be seen as the significant contributions of the present investigation.

- Performance of helium purifier with various concentrations of impurities gives grade 4.5 helium.
- Coconut shell activated charcoal has worked fine as those being virgin, but we need to observe its performance in long run if purifier is run regularly for one year.
- Performance analysis of heat exchanger has been done.
- This purifier can be used commercially for purifying helium upto 40% impurity

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