Development and Study of a Helium Purifier based on Low Temperature High Pressure Adsorption of Impurities

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

Master of Technology (Research)

in

Mechanical Engineering

By

Trijit Kumar Maiti

Department of Mechanical Engineering

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Under The Guidance of

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Department of Mechanical Engineering

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NATIONAL INSTITUTE OF TECHNOLOGY

ROURKELA

January 4, 2011

CERTIFICATE

This is to certify that the thesis entitled "Development and Study of a Helium Purifier based on Low Temperature High Pressure Adsorption of Impurities", being submitted by Shri Trijit Kumar Maiti, is a record of bona fide research carried out by him at the Department of Mechanical Engineering, National Institute of Technology, Rourkela, under our guidance and supervision. The work incorporated in this thesis has not been, to the best of our knowledge, submitted to any other university or institute for the award of any degree or diploma.

(Sunil K. Sarangi) (Ranadhir Dey)

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(Trijit Kumar Maiti)

ABSTRACT

Most helium liquefiers today operate on modified Claude cycle with at least two high speed turbo expanders. Impure gas cannot be fed to these liquefiers as air impurities solidify at low temperature, and damage the turbo expanders and choke the tubes. 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% helium to the liquefier after separating air contaminants from impure helium. At present, research and academic centres in India, import helium purifiers which are very expensive in terms of foreign exchange. National Institute of Technology, Rourkela, has taken up a project for development of cryosorption based helium purifier funded by the Board of Research in Nuclear Sciences, Mumbai, which primarily aims at developing helium purifier and studying its performance. This dissertation details the design, fabrication and performance analysis of a prototype helium purifier. The purifier is based on the principle of cryocondensation of moisture and air impurities on heat exchangers at appropriate temperatures and cryosorption on activated charcoal to yield Grade 4.5 helium from 60% pure helium at LN_2 temperature. The purifier has been designed for 6 hours nonstop operation with throughput of 20 nm^3/hr and delivery of pure helium at a pressure of 150 bar(a), which is ensured by a reciprocating compressor. The vital component, adsorber columns, was packed with Indian manufactured granular coconut shell activated charcoal in 50 NB Sch 80 SS 316L pipe. Other major components include moisture collector vessel, liquid air separator vessel, three heat exchangers, snow filter, gas bag, cylinder manifold and superinsulated LN_2 vessel housing all cryogenic components of purifier. Helium gas bag, made of textile reinforced rubber has been successfully developed. All the components were fabricated, assembled and commissioned at NIT, Rourkela. Conditioning of the system was carried out by heating with six 1000 W tubular heaters, followed by evacuation. The system was back filled with Grade 4.5 helium. Experiment was carried out for one session by feeding the purifier with 5% dry nitrogen contaminant in helium as input gas, and four samples of purified gas were collected in sample cylinders at different intervals. Sample analysis by Linde Multi Component Detector reveals that the total impurity, consisting of nitrogen, oxygen and moisture, is less than 5 ppm by volume, thus making the purified gas much better than Grade 4.5 helium.

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Chapter 1

INTRODUCTION

Helium, because of its unique properties, stands out to be indispensable in frontier technologies especially in the domain of space, atomic energy, defence and medical sciences. The single largest application of pure helium is in cooling superconducting magnets, key to high energy accelerators, superconducting cyclotron and Magnetic Resonance Imaging (MRI) scanners. In India, helium is an expensive and imported consumable. Therefore, a helium purifier is an integral part of any cryogenic centre to conserve helium gas.

The basic theories regarding cryosorption based gas purification are well established and are widely available in open literature, but the finer aspects of technology still remain proprietary information. The work reported in this thesis is an attempt to design, construct and commission a helium purifier through experimentation.

1.1 Function of Helium Purifier

In a large cryogenic installation, generally, helium is maintained in closed loop cycle. But, due to failure of the system or any of its components, the cold gas or liquid helium of the system expands to large volume and causes pressurization, resulting in blow off of safety relief valve. Because of the high cost and limited availability, helium is recovered in gas bag which causes ingression of air and moisture contaminants by diffusion. Similarly, laboratory experiments with cryostat and superconducting magnet, generates helium gas which is collected in helium gas bags. Recovery compressor compresses this impure gas which results in oil contamination in high pressure gas storage. All these impurities are required to be eliminated to obtain Grade 4.5 helium gas suitable for running a liquefier. Hence, a helium purifier is an indispensable device in any cryogenic set up in India.

1.2 Principles of Helium Purification

The basic principles involved in helium purification process are condensation of air impurities on heat exchangers and cryosorption of remaining impurities over activated charcoal columns at 120 bar(a) or higher at 77 K [2][12][29]. The purifier system has been designed to purify 60% pure helium to 99.995% purity. Gas bag

collects impure helium from cryogenic experiments or from high pressure gas storage system, and acts as low pressure buffer for compressor inlet. The high pressure is helpful in reducing the fraction of impurities in the process stream before it enters the adsorber columns, because the total pressure is high while the partial pressure of impurities is limited to the saturation pressure at 77 K. A high pressure compressor compresses this impure helium to 120 bar(a), or higher up to 150 bar(a) at a flow rate of 20 nm^3/hr. The gas then passes through the oil and water separator vessel, three heat exchangers and liquid air separator vessel in sequence. The first heat exchanger acts as dryer which brings down the water dew point to approximately 1^oC to facilitate liquid drainage. The second and the third ones condense air impurities and reduce the partial pressure below 1 atmosphere, following the principles of vapour-liquid equilibria. Liquid air separator vessel then collects this condensed air with some floating water and CO_2 ice which is blown down at regular intervals depending on the impurity level. After that, a snow filter filters any solid CO_2 or ice coming with the impure stream. Now, helium, containing less than 0.8% air impurity, enters the adsorber columns which reduce air impurity to less than 50 ppm and thus yields Grade 4.5 helium. Normally imported helium purifiers use anthracite coal based charcoal as cryo-adsorber, but we have used indigenously available coconut shell based activated charcoal. The third heat exchanger, liquid air separator and adsorber columns are totally submerged in LN_2. A back pressure regulator at the outlet maintains a minimum operating pressure of 120 bar(a) over adsorber columns during the flow period. The helium purifier works in two phases – Purification and Regeneration. After completion of purification phase, LN_2 is drained out and adsorber columns undergo regeneration by heating and evacuation.

1.3 Objectives of the Work

At present, academic institutions and research centres of India import helium purifiers which are not only very expensive in terms of foreign exchange but also are costly to maintain. Indigenous development will not only make the device available at low cost but will enhance maintainability in short notice. National Institute of Technology, Rourkela has taken up the project of development of cryogenic adsorption based helium purifier in collaboration with Variable Energy Cyclotron Centre, DAE, Kolkata. The objectives of this work are as follows:

(i) Design, development, installation and commissioning of cryosorption based helium purifier and its performance analysis under varying input parameters.

(ii) Proper documentation for future construction on commercial basis and technology transfer.

(iii) Development of components of purifier in India in collaboration with private manufacturers i.e. vendor development in India for cryogenic technology.

1.4 Organization of the Thesis

The thesis has been arranged in seven chapters and appendices. Chapter 1 deals with a general introduction to the helium purifier and defines the scope of the work. Chapter 2 presents a brief review of the literature, covering the experimental techniques of using different types of adsorbents at cryo-temperature for impurity adsorption from helium stream, techniques to obtain adsorption isotherms of activated charcoal at different cryogenic temperatures and pressures. This review provides the information regarding current research and the performance of the large capacity cryogen based helium purifiers already installed at several cryogenic centres throughout the globe. Chapter 3 illustrates the specification and function of each component of the purifier, basic criteria of adsorbent selection and developmental procedure of helium gas bag. The design procedure of adsorber columns, LN_2 vessel of purifier and tubular heaters have been presented in this chapter. Chapter 4 highlights the processes involved in the fabrication of the constituents of purifier and their assembly, while chapter 5 describes the experimental set up to study the performance of the helium purifier and its operation procedure. Experimental results of the prototype helium purifier have also been included in this chapter. Chapter 6 presents concluding remarks and recommendation for future work. And finally a list of references is presented which future workers on the subject will find useful. This thesis contains three appendices which record specification of components, design and fabrication drawings, which can guide future workers.

Chapter 2

LITERATURE REVIEW

In the field of cryogenic technology, an engineer is concerned with the development and utilization of processes, instruments and devices utilizing the low temperature principles. In the field of gas separation and purification, fundamental principles, design, construction procedures have been discussed in well known text books and hand books on cryogenic engineering [1-7]. The books [1][2][6] provide introduction to the field of cryogenic engineering and contain the fundamental data on low temperature adsorption systems.

2.1 History of Development of Gas Purification System

The world's first air separation plant for the production of oxygen was established by the Linde Group in 1902 and in 1913 they made a successful attempt in installing world's first recovery of rare gases on a commercial scale [41]. In 1917, the Bureau of Mines, USA, in collaboration with Linde Company, Air Reduction Company and Jefferies-Norton Corporation, installed three experimental plants at Texas, USA, to extract helium from natural gas [2]. In 1989, the Linde Group installed the largest pressure swing adsorption plant in Europe with vacuum regeneration for production of oxygen from air [41]. Today, in USA, helium recovered from natural gas, is liquefied in large scale industrial refrigerators and a part of this product is shipped to other countries in liquid form instead of high pressure gas cylinders [1].

2.2 Gas Purification by High Pressure and Low Temperature Adsorption

The design and development of gas purification system is usually the result of several engineering disciplines: Cryogenic Engineering, Thermal analysis, piping/tubing system, adsorption technology, vacuum engineering, welding technology, controls, mechanical design and gas composition analysis. The book, Gas Purification by Kohl and Riesenfeld [23], provides a thorough, authoritative engineering treatment on the gas purification and dehydration processes most commonly used in industry. Basmadjian [7] addresses the phenomenon of adsorption process and also provides theoretical analysis

on preliminary sizing of an adsorber bed, estimation of bed and purge requirement. Haselden [6] presents the outline of designing adsorber vessels for both pressure and thermal swing adsorption. Perry's handbook [22] is a very effective tool for selection of adsorbent. It provides the minute details and characteristics of several types of activated charcoal available in the market. Yang et. al. [15] summarise the experimental data of adsorption isotherms on coconut shell activated charcoal in the low and medium pressure range of 3.45 x 10^3 to 2.07 x 10^6 N/m^2 and temperature range of 150 K to 300 K. The dimensions and constituents of adsorption column, the adsorbent used for the experiment and the experiment set up have been discussed by the authors. 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. [17] 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). Leyarovski et. al. [33] have studied the desorption of cryogenic gases from activated charcoal at low temperature. Their results have lead to reduction of electrical power and LN_2 consumption in large sized cryogen based purifiers. The authors have concluded that due to low temperature regeneration, certain grade of activated charcoal saturated with nitrogen and oxygen could be reactivated at LN_2 temperature up to 30% to 40%.

Thingstad [26] have explained in brief about the cryogenic purifier installed for streamer chamber at Stanford Linear Accelerator Center, USA. This purifier was used for purifying neon-helium mixture with impurities of 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.

Stoll et. al. [27] commissioned a fully automatic large capacity helium purifier with operating pressure of 150 bar(g), 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, recooling and repressurization 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.

Neumann et. al [12] have described the helium purifier manufactured in 1996 by L' Air Liquide, France, a leading cryogenic technology provider to the world. This purification plant was operated at 200 bar(g) pressure with flow rate of 14 gm/s, the continuous operation time being 12 hours. The impurities handled were air and oil impurities normally encountered in cryogenic experiments, temporary storage and use of recovery compressor. Oil impurity was removed by two coalescing filters in series followed by active charcoal filled adsorber beds at room temperature. In the second stage moisture was eliminated by molecular sieve adsorber, and finally, in the third stage removal of nitrogen and oxygen took place to get pure helium. The adsorbent used was a special type of silica gel dipped in LN_2. The regeneration steps included removal of LN_2 and heating the adsorber bed to 200 K before evacuating and pressurizing with pure helium for inerting. A multiple component detector from Linde AG, Germany was used for analysis of the output helium. The purified helium had impurity content of less than 1 ppm.

Richardson et. al [13] constructed a neon purification system at Fermilab, USA. They used coconut shell activated charcoal as adsorbent at LN_2 temperature to remove nitrogen, oxygen, CO_2 and moisture. Design data of the adsorber and adsorber characteristics have been presented in their paper. Their system consisted of a 15 hp gas compressor, a SS 304 vessel containing 64 kg of activated charcoal, four 600 W heaters, freezer, piping system, heat exchanger, instrumentation and an LN_2 jacket to maintain cryogenic temperature at adsorbent bed. The system operated at 9.6 bar(g) pressure with bed pressure drop of 1.2 bar at volumetric flow rate of 1529 l/min. Output neon had nitrogen impurity of less than 100 ppm when input nitrogen contamination was 3%.

Max Planck Institute, Germany, [30] installed a helium purification system for their project on fusion experiment W7-X. It consisted of two compressors and cryogenic adsorber beds operating at LN_2 temperature and a pressure of 20 bar(g). Its purification capacity was 2.5 gm/s for 5% dry air content and moisture impurity corresponding to saturation at 25^{o}C, operating time being 12 hours purification time for one line.

Experiment was carried out with a small model cryopurifier at VECC, Kolkata, India [29]. 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.

KEK, Japan, [32] has set up a large off-line helium purification unit consisting of reciprocating compressor operating at 150 bar(g) with flow rate of 150 nm^3/hr and adsorption beds operating at 80 K.

In India, a project [34] on extraction of helium from natural gas was taken up by the Department of Atomic Energy and Department of Science and Technology. By employing 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.

2.3 Safety Issues associated with Cryogenic Purifiers

One incident [35] of failure says that explosion occurred in a charcoal bed of a helium purifier while operating with 1.40% oxygen and 0.70% nitrogen impurity at 200 bar. This took place when the operator opened a new cylinder manifold for charging. It was found that oxygen of purity above 85% soaked the charcoal which got ignited due to flow and pressure surge when the helium was switched from full to empty manifold. Fragments and parts were found up to 250 m away. Another incident occurred in a laboratory while regenerating. The purifier had been operating for 2 to 3 days with 5% air impurity. When the system was electrically heated during reactivation, the explosion occurred which damaged the building structure. Examination found that excessive high temperature produced due to charcoal and oxygen reaction was the probable cause of explosion.

The abovementioned systems were equipped with safety relief devices, but the reaction was so rapid that the conventional safety devices could not save the system. So, some of the following measures are suggested while designing a system. The oxygen content of the impure process gas must be measured and limited so that condensation of liquid oxygen be checked in charcoal adsorber vessel. Sudden flow or pressure surge through activated charcoal bed be avoided to prevent dusting of charcoal and likelihood of ignition. Regeneration system should be such that at any point temperature of charcoal must not exceed the designed value.

Chapter 3

PROCESS DESIGN OF HELIUM PURIFIER PROTOTYPE

3.1 Technical Specification of Helium Purifier Prototype

The basic input parameters of the helium purifier are the following:

Flow rate	: 20 nm^3/hr (approximate)
Delivery pressure	: 150 bar(a)
Input gas purity	: 60% i.e. 40% air impurity (maximum)
Output gas purity	: 99.995% (minimum) i.e. Grade 4.5, i.e. max. allowable impurity is 50 ppm by volume
Run time for purification	: 6 hours
Regeneration process	: Heating and evacuation
Type of heater	: Nichrome element based tubular heater
Adsorbent	: Coconut shell granular activated charcoal
Regeneration temperature	: 120^oC
Operating temperature	: LN_2 temperature i.e. 77 K

Piping and Instrumentation Diagram (P&I Diagram) of the helium purifier is presented in Fig. 3.1.

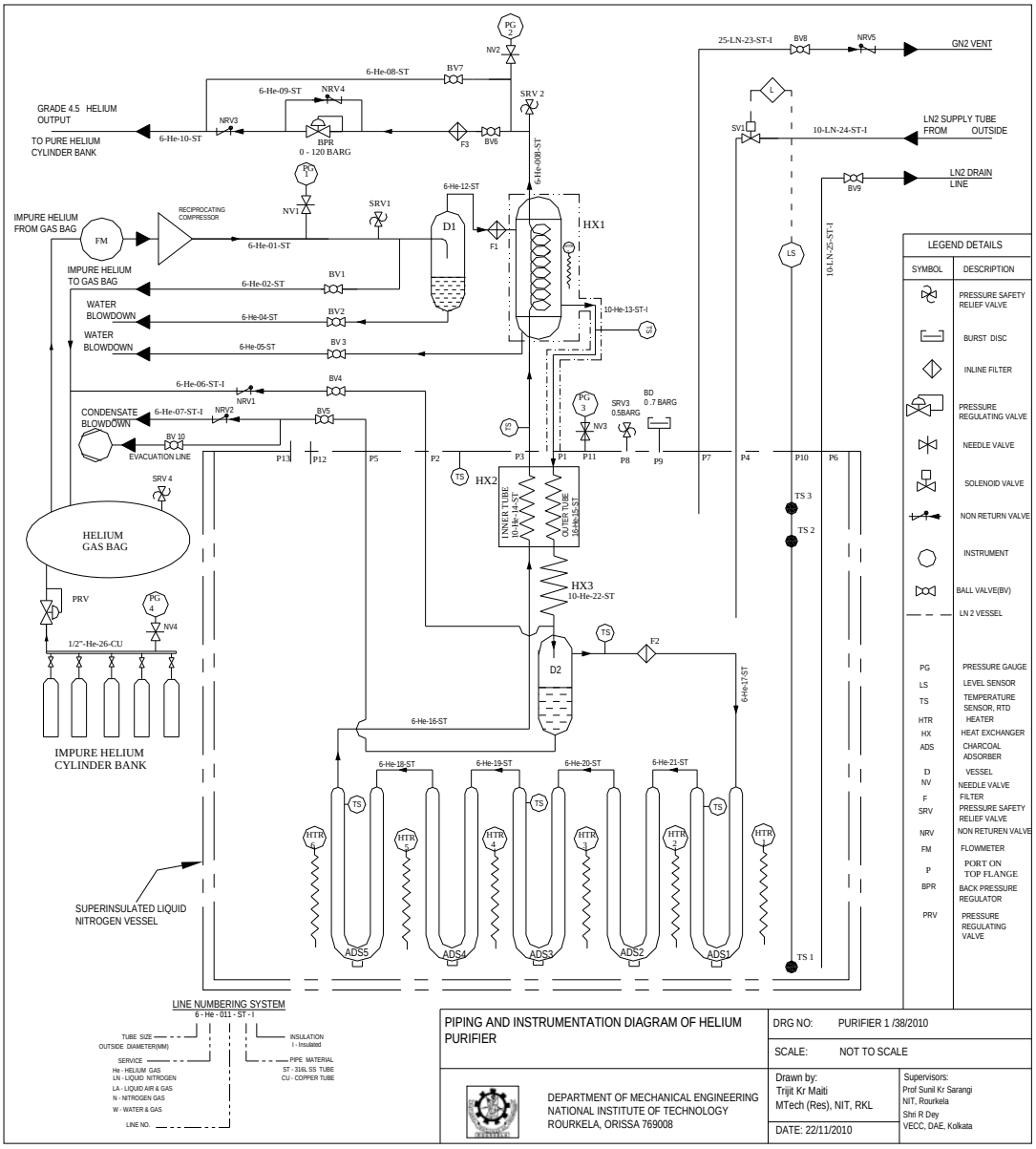

Fig 3.1: P & I Diagram of helium purifier

3.2 Description and Utility of Principal Components of Purifier

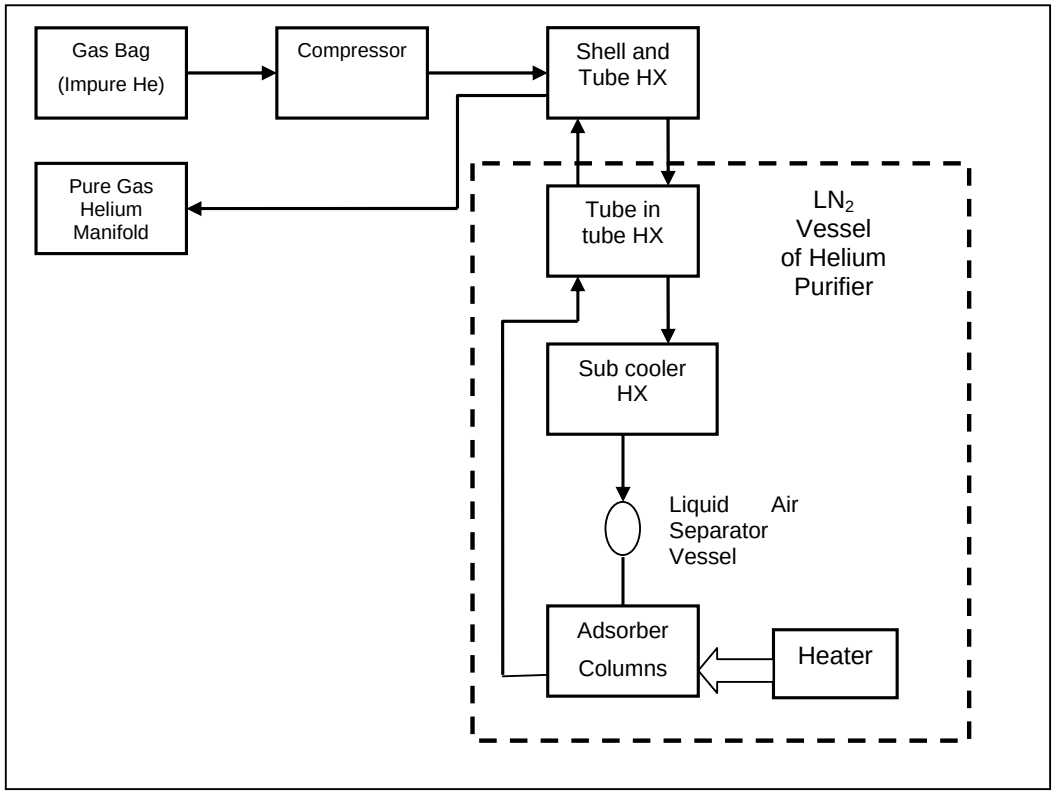

Fig. 3.2: Block diagram of helium purifier system

The Principal Components of Purifier

The principal components of helium purifier system are the following [refer Fig. 3.1 for nomenclature]:

(1) Gas Bag, (2) Compressor, (3) Moisture Separator Vessel, D1, (4) Shell & Tube Heat Exchanger, HX1, (5) Tube-in-Tube Heat Exchanger, HX2, (6) Subcooler Heat Exchanger, HX3, (7) Liquid Air Separator Vessel, D2, (8) Adsorber Columns, ADS, (9) Tubular Heaters, HTR, (10) Snow Filter, F2, (11) LN_2 Vessel of Helium Purifier, housing Tube-in-tube exchange, Subcooler, Liquid Air Separator Vessel and tubular heaters (12) Back Pressure Regulator, BPR, (13) Pure and Impure Helium Cylinder Manifold, (14) Pipe network consisting of tubes, valves and gauges, (15) Instrumentation, (16) Linde Multi-Component Detector for purity checking.

Gas Bag

The gas bag operates at a pressure of 50 mbar(g) and acts as a buffer at the compressor inlet. It functions as temporary storage of impure helium coming from impure manifold or from an experimental set up. It is made of rubberised fabric with low permeability of helium and the joints are carefully made to avoid leakage.

Compressor

A three stage reciprocating type high pressure helium compressor takes impure helium from gas bag and compresses to 150 bar pressure and delivers to purifier at flow rate of 20 m^3/hr. This is a specially sealed machine which has very low helium leak rate, of the order of 10^{-4} mbar-l/s. The delivery pressure starts from a low value equal to the receiver cylinder bank pressure and rises with time.

Moisture Separator Vessel

This vessel is situated after the compressor and acts as accumulation place for condensed moisture and oil from compressor outlet gas. As the compressor is oil lubricated, so the outflow helium gas from compressor contains moisture and lubricating oil. These get separated at this vessel. The periodical blow down is done to ensure condensate free gas entering the purifier.

Shell and Tube Heat Exchanger

Shell and tube heat exchanger, is a counter-current heat exchanger where coiled tube is bundled inside the shell. The heat exchanger, located after moisture separator vessel, acts as a bulk eliminator of moisture from impure helium. Cold pure helium runs through the tube and impure helium at normal temperature flows inside the shell over the coiled tube. The moisture gets condensed on the tube surface and is collected within the shell which is removed periodically by opening the drain valve.

Tube-in-Tube Heat Exchanger

This is tube-in-tube helically coiled counter current flow heat exchanger made of 16 mm OD and 10 mm OD SS 316 tubes. This is located above the maximum LN_2 level within LN_2 vessel. Due to the reasons of high efficiency, easy to fabricate, compactness, this type of heat exchanger has been selected as cryogenic heat exchanger.

Subcooler Heat Exchanger

This is helically coiled heat exchanger and is fully submerged in LN_2 within LN_2 vessel. This functions as subcooler, where nitrogen impurity gets condensed at operating pressure and is collected at liquid air separator vessel.

Liquid Air Separator Vessel

Liquid air separator vessel is located within LN_2 vessel and is submerged in LN_2. This vessel acts as phase separator where condensed air from cold 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. The helium gas escapes through the port at the top of the vessel. The condensate is blown down periodically.

Adsorber Columns

Adsorber columns are fully submerged in LN_2 within LN_2 vessel. The columns are U-shaped vertical stainless steel columns and five in number, connected in series. All the adsorber columns are similar in shape and size. Coconut shell based activated charcoal adsorbent is tightly packed in the columns. Adsorption of air and moisture impurities takes place in adsorber columns at 77 K and 120 bar(a) pressure or higher. This is ensured by keeping a back pressure regulator set to 120 bar(a) after the adsorber columns.

Tubular Heater

Tubular heaters are fixed within LN_2 vessel. These Nichrome wire based heaters heat up the heat exchangers, liquid air separator, snow filter and adsorber columns for desorption of air and moisture contaminants during regeneration phase of purifier. Heaters heat up purifier components from 77 K to 393 K.

LN_2 Vessel of Purifier

This is superinsulated LN_2 vessel housing all the cryogenic components like heat exchangers, adsorber bed, liquid air separator vessel, heaters and LN_2 level indicator. 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 arranged in a

cage which is suspended from top flange. All the process connections and electrical feedthroughs pass through the top flange.

Back Pressure Regulator

Back pressure regulator controls the pressure upstream of the delivery port. This device operates like a sensitive, externally adjustable relief valve. This regulator maintains the adsorber columns pressure stably at 120 bar(a). During the purification stage it reduces the adsorbate load on the charcoal columns.

Cylinder Manifold

There are two cylinder manifolds for storing high pressure pure and impure helium gas. In pure gas manifold, purified gas is collected at pressure of 150 bar from the purifier. Impure manifold stores impure gas collected from various external sources viz. cryogenic experimental set up, etc.

3.3 Development of Helium Gas Bag

The gas bag acts as a temporary storage for impure helium gas at low pressure. Presently, liquid helium user establishments of India import helium gas bags which are very expensive, need long delivery time and are difficult to maintain. So, we made an extensive market survey for developing special gas bag indigenously and finally collaborated with Softex Industrial Products Pvt. Ltd, Kolkata, India to develop helium gas bag.

Initially, a 1 m^3 sample gas bag was fabricated and pressure test was carried out both at manufacturer's site and the laboratory at NIT, Rourkela. Based on satisfactory results of the sample gas bag, a 5 m^3 helium gas bag was fabricated, pressure tested and installed at laboratory for helium purifier.

The reputed suppliers of helium gas bag worldwide are M/s Air Liquide, France, M/s Linde Process Plants, USA, M/s Flexi-Liner Corporation, USA. M/s Air Liquide supplies gas bag made of Polyester 550 dtex textile with PVC coating on two sides and air permeability less than 2 $l/m^2/24$ hr [37]. M/s Flexi-Liner Corporation used to manufacture Eurothane elastomeric helium recovery gas bags.

Fig. 3.3: Helium gas bag installed at the laboratory

Technical Details of 5 m^3 Helium Gas Bag

The detailed technical specification of the gas bag is the following:

(1) Length and width of 5 m^3 gas bag in deflated condition are 3 m and 2.5 m respectively, and diameter in inflated condition is 1.6 m. Total weight is 18 kg.

(2) Physical Characteristics of Gas Bag Material

The material of construction of gas bag is textile reinforced rubber, which has 4 layers viz. chlorosulphonated 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 $\pm$ 0.10 mm. Fig. 3.4 shows the multiple layers of gas bag material. Dimensions are in mm in all the drawings of this dissertation.

Other physical properties of gas bag material are as follows:

- Breaking Strength (kg / 20 mm width)
  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:1100 g/m^2

The material is resistant to fungi, corrosion, most organic solvents like ketones, alcohols, gasoline, oil, etc and weathering. It also has excellent retention of

physical and chemical properties at low temperatures including cold abrasion resistance, etc.

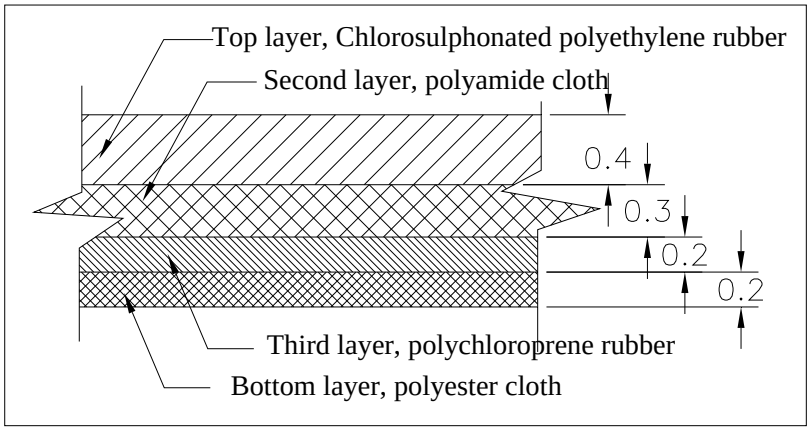

Fig. 3.4: Material of construction of gas bag

(3) Operating pressure: 50 mbar(g)

(4) Width of the rubber cloth used for gas bag is 1280 mm

(5) Two hollow cloth loops of proper size on each side of gas bag for fastening with gas bag holding structure, and one loop at the top to support float for limit switch, i.e., total of 5 loops are there.

(6) Construction Details of Gas Bag Nozzle

The gas bag nozzle acts as an entry point of gas from outside sources or from cylinder manifold. Gas is also delivered to compressor through this nozzle via flexible braided hose.

The gas bag nozzle is a critical element, so proper precautions were taken during its fabrication. Gas bag has only one nozzle and nozzle pipe size is 25 mm outer diameter. Nozzle consists of one nozzle pipe and two flanges, one inside the gas bag and the other outside. The flanges are injection molded class 150 flanges made of PTFE. The inside flange is joined by threads on the nozzle pipe. The bolts used in flange are of stainless steel (AISI 304) and have size M10. PVC braided hose of 25 mm inner diameter is clamped over the nozzle pipe. Neoprene rubber washers have been used between flange and cloth on both inside and outside for sealing. The inside flange is 25 mm thick and have tap depth of at least 15 mm. Fig. 3.5 shows the details of the nozzle of gas bag.

(7) Installation of Helium Gas Bag

This gas bag is suspended from the ceiling of the laboratory by mild steel structure. Light weight mild steel pipes of 25 mm NB are anchored to the ceiling by anchor bolts. The gas enters and going out of the gas bag through the flexible hose connected to nozzle. Fig. 3.3 shows gas bag installed at the laboratory at NIT, Rourkela. The schematic diagram of the gas bag system is shown in Fig. A10 of appendix II.

Test at Manufacturer's Works

Initially, the helium gas bag was evacuated for half an hour by rotary vacuum pump for removal of air and moisture. Then it was filled with dry compressed air to raise pressure up to 200 mbar(g) at 34°C ambient temperature. The conditions inside the gas bag pressure was observed hourly with both pressure as well as temperature noted. Gas pressure was measured by Bourdon type gauge with the least count of 0.50 mbar. After 79 hours, the pressure drop was found to be 2.50 mbar at fixed ambient temperature. The pressure drop was negligible, so the product is satisfactory and acceptable.

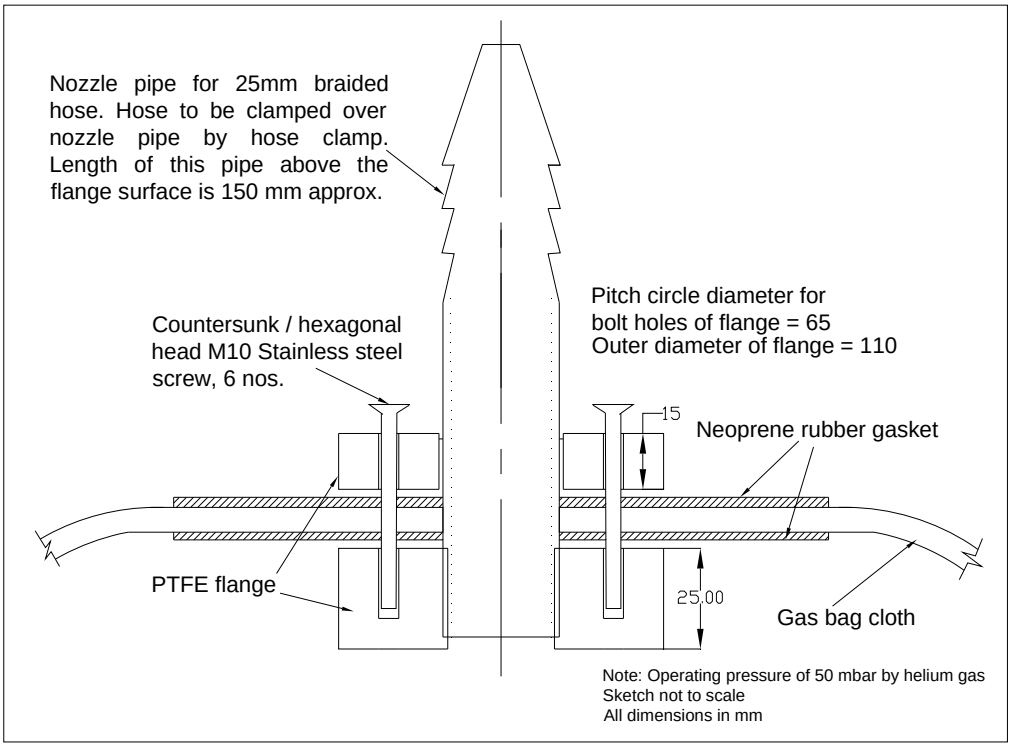

Fig. 3.5: Nozzle of the flange of helium gas bag

3.4 Selection of Helium Compressor

A compressor is necessary for creating the high pressure of 150 bar(a) to ensure efficient condensation and adsorption of impurities. 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 from various sources. So, extensive market survey has been conducted on high pressure compressors with various manufacturers in Asia, US and European countries. The European and USA make compressors with good reputation in international market turned out to be too expensive. So we have chosen Metec Corporation, South Korea, which is cheaper than any USA or European model.

3.4.1 Technical Specification of Helium Compressor

Model No.: Series 1000B - He

- Flow capacity: 20 nm^3/hr
- Inlet gas pressure: less than 0.5 bar(g)
- Operating pressure: 150 bar(a)
- High pressure switch pressure setting: 155 bar(a)
- Safety valve pressure setting: 160 bar(a)
- Discharge valve outlet port: ¼”
- Motor power: 7.50 kW, 400 VAC, 50 Hz
- Oil removal unit consists of two micron filters in series viz. 1 micron and 0.01 micron with auto drain valve.

Fig. 3.6 shows the photograph of the compressor installed at laboratory for helium purifier.

Fig. 3.6: Reciprocating compressor

3.5 Design of Moisture Separator Vessel

Moisture separator vessel is a gas-liquid vertical cyclone separator. For design purpose, input impure helium is considered to be saturated with moisture at 300 K. The flow rate of impure helium is 20 nm^3/hr, partial pressure of saturated moisture is 35.70 mbar and specific volume is 38.78 m^3/kg at 300 K [39].

Moisture content of impure helium

= (20 / 38.78) kg/hr

= 0.51 kg/hr.

After compressing to 150 bar(a) at 300 K, helium flow rate becomes (20 / 150) m^3/hr = 0.13 m^3/hr and helium is saturated with moisture, and the moisture content

= (0.13 / 38.78) kg/hr

= 3.35×10^{-3} kg/hr.

Moisture condensed in moisture separator vessel

= $(0.51 - 3.35 \times 10^{-3})$ kg/hr

= 0.507 kg/hr

= 5.07×10^{-4} m^3/hr of water

Let us choose pipe size 50 NB Sch 80 (OD = 60.3 mm, thickness = 5.54 mm) and let the length of pipe required be 'L' m for condensate collection in 30 minutes. Equating

$(\pi/4) \times 0.04922^2 \times L = (5.07 \times 10^{-4} / 2)$

or, L = 0.133 m

For cyclonic movement of inlet gas, minimum pipe length requirement [22]

= 2 x Inside diameter of pipe

= 2 x 0.04922 m

= 0.09844 m

Hence, minimum pipe length required below inlet

= (0.133 + 0.09844) m

= 0.23 m

Adding height of top end cap (0.038 m) and extra space above inlet, the final external length of moisture separator vessel comes out to be 0.3 m. During operation, condensate should be blown down at maximum interval of 30 min.

3.6 Design of Liquid Air Separator Vessel

Liquid air separator vessel is a vertical cyclone separator located inside LN_2 vessel after heat exchangers. For design purpose, let us consider 40% nitrogen impurity in

impure helium at delivery pressure 150 bar and volumetric flow rate 20 nm^3/hr. Ambient temperature is 300 K. After cool down of impure helium at heat exchangers, the amount of nitrogen remains in helium stream after liquid air separator vessel is only 0.67% [refer subsection 3.7.3 for detailed calculation]. Volumetric ratio of nitrogen at 300 K to LN_2 is 707.

Amount of nitrogen gas separated from helium stream in liquid air separator vessel

= (40 − 0.67)% of 20 nm^3/hr

= 39.33% of 20 nm^3/hr

= [(20 x 39.33) / (100 x 707 x 3600)] m^3/s of LN_2

= 3.09 x 10^{-6} m^3/s of LN_2

Dimensions of Liquid Air Separator Vessel

Let us choose duration of 15 min as minimum blow down interval of liquid air separator vessel and select SS pipe of size 100 NB Sch 80 pipe (OD = 114.30 mm, thickness = 8.56 mm i.e. ID = 0.097 m) to optimise the sizing of liquid air separator and LN_2 vessel. Let 'h' m be length of pipe required to collect LN_2 condensate.

Volume of LN_2 produced in 15 min = 2.78 x 10^{-3} m^3

Therefore,

2.78 x 10^{-3} = (π/4) x $(0.097)^2$ x h

Or, h = 0.37 m

Pipe length required for cyclonic movement of gas [22]

= 2 x inside diameter

= 2 x 0.097 m

= 0.194 m

Minimum length of vessel below inlet

= (0.37 + 0.194) = 0.564 m

Adding height of top end cap (64 mm) and extra space above inlet, final length of liquid air separator vessel comes out to be 0.64 m.

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

$u_t = 0.07 \sqrt{[(\rho_L - \rho_v) / \rho_v)]}$

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 = Gas density, kg/m^3

= Density of helium at 150 bar(a) and 77 K = 70 kg/m^3

Applying the formula,

u_t = 0.07 √ [(813.89 – 70) / 70]

= 0.23 m/s

The diameter of vertical gas-liquid cyclone separator should be large enough to slow the gas down to below the velocity at which the liquid droplets will settle out. The minimum allowable vessel diameter, D_v, is expressed [38] as

D_v = √ [(4V_v) / (π x 0.15 x u_t)]

Where,

V_v, Helium flow rate at 77 K and 150 bar(a) = 9.44 x 10^{-6} m^3/s

Hence,

D_v = √ [(4 x 9.44 x 10^{-6}) / (π x 0.15 x 0.23)]

= 0.0187 m

The result validates selection of 100 NB Sch 80 SS pipe.

Deriving Maximum Blow Down Interval

Condensate should be blown down from liquid air separator at proper time interval, otherwise it will overflow and enter adsorber columns and saturate it. Following calculation shows the maximum blow down time interval of the vessel when helium bearing 40% nitrogen impurity flowing at rate 20 nm^3/hr at 150 bar pressure.

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

Maximum blow down interval

= (LN_2 accumulation in 15 min / LN_2 formation rate)

= [(2.78 x 10^{-3} / 3.09 x 10^{-6}) / 60] min

= 15 min

Table 3.1 shows the maximum blow down interval of liquid air separator vessel with respect to nitrogen impurity concentration in helium flow. It is found that below 20% nitrogen impurity blow down interval is half-an-hour.

Table 3.1: Variation of blow down interval with nitrogen impurity concentration

Sl	Nitrogen impurity concentration (%)	Maximum blow down interval (min)
1	1	1754.5
2	2	435.3
3	3	248.5
4	4	173.87
5	5	133.7
6	10	62
7	15	40
8	20	29.9
9	25	23.8
10	30	19.7
11	35	16.8
12	40	15

Design for Non-re-entrainment of LN_2 with Outgoing Helium

In cyclone separator, re-entrainment of condensate in outgoing helium is generally reduced by lowering gas velocity. Onset of entrainment occurs at cyclone inlet velocity V_{ci}, m/s, in accordance with the relationship [22],

$V_{ci} = 6.516 - 0.2865 \ln (N_{Re,L})$

Liquid Reynolds number on the wall of the vessel, $N_{Re,L} = 4Q_L / (h_i \nu)$

Where,

Kinematic viscosity of LN_2 at 77 K, 1 bar(a), $\nu = 1.93 \times 10^{-7}$ m^2/s

Volumetric LN_2 flow rate, $Q_L = 3.09 \times 10^{-6}$ m^3/s

Inlet height for cyclonic movement of gas, $h_i = 0.194$ m

Flow rate of helium at 150 bar(a) and 77 K = 0.034 m^3/hr

Gas inlet tube of vessel is 10 mm OD x 1 mm thick, so inner diameter is 0.008 m

Entry velocity of helium in liquid air separator vessel

$= (0.034) / [\{\pi (0.008)^2 / 4\} \times 3600]$ m/s

= 0.18 m/s

Liquid Reynolds number on the wall of the vessel, $N_{Re,L}$

$= (4 \times 3.09 \times 10^{-6}) / (0.194 \times 1.93 \times 10^{-7})$

= 330

Hence, $V_{ci} = 6.516 - 0.2865 \ln (330) = 4.85$ m/s

As entry velocity of helium is less than $V_{ci,}$, re-entraiment of LN_2 in exit flow will not occur.

3.7 Design of Adsorber Columns

Adsorption is a separation process where a fluid (gaseous or liquid) comes in contact with a porous particulate phase and with property of selectivity, one or more species in fluid phase gets transferred to solid porous surface. It is often the most economical method of separation if the constituents to be adsorbed are present at relatively low concentrations.

3.7.1 Selection of Suitable Adsorbent for Helium Purifier

The adsorbents commonly available in Indian market are Activated Alumina, Molecular Sieve or Zeolite, Silica Gel and Activated Charcoal.

Selection of Adsorbent for Cryosorption

Activated charcoal is of various types based on their origin, viz. wood based, coconut shell based, lignite based, peat based, bituminous coal based, anthracite coal based, petroleum based, etc. Coconut shell based activated charcoal is having maximum BET surface area and less bulk density compared to other available adsorbents. More BET surface area means more micro-pores for multilayered gas adsorption. The lesser bulk density of activated charcoal reduces the adsorber columns 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 [14][15][38] of activated charcoal is better than molecular sieve, commercially available 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. Yang et. al. [15] provides limiting adsorption mass fraction data of nitrogen on molecular sieve 13X, granular GI activated carbon and compressed GI activated carbon. The values of limiting adsorption mass fraction obtained by the method of liquid nitrogen adsorption at 78.92 for the abovementioned adsorbents are 0.141, 0.489 and 0.456 respectively. Fig. 3.7 shows the comparative study of adsorption isotherms [15] of nitrogen on molecular sieves 13X and granular GI grade activated carbon, expressed in mass of nitrogen gas adsorbed per unit mass of adsorbent, (Δ m)/M, i.e., the mass fraction as a function of gas pressure.

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

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 of activated charcoal available to us from the manufacturer is shown below in Table 3.2. BET surface area has been measured in the institute.

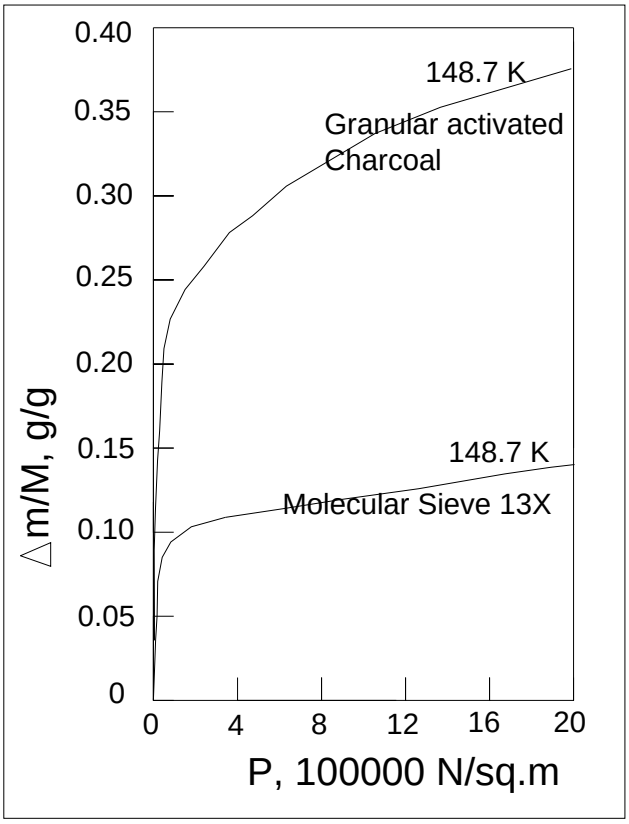

Fig. 3.7: Nitrogen adsorption isotherms on molecular sieve and activated charcoal

Table 3.2: Technical data sheet of activated charcoal grade AC 4/8

Characteristics	**Property of charcoal**
Make	M/s Exal Corporation, Vadodara, Gujrat, 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

Characteristics	Property of charcoal
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, kg/m^3	550

3.7.2 Manufacturers and Suppliers of Activated Charcoal in India and Abroad

M/s Calgon Carbon Corporation, Pittsburgh, USA is one of the most reputed global manufacturer and supplier of activated carbon. This manufacturer produces different types of granular, powdered or pelleted activated charcoal from coal, wood or coconut shell. Another pioneer global supplier is M/s Norit Active Carbons, a Netherlands based company. India is one of the major exporters of coconut shell charcoal which are generally used for effluent water treatment, catalytic reaction in chemical and petroleum refinery, purification of drinking water and industrial gases and pharmaceuticals & bulk drug industries. The reputed manufacturers in India are M/s Exal Corporation, Vadodara, M/s Indo German Carbons Ltd, Kerala, M/s Active Carbon India Pvt. Ltd, Hyderabad, M/s Industrial Carbons Pvt. Ltd, Ankaleshwar, M/s Western Chemical Corporation, Vadodara, and so on. These Indian companies produce activated carbon from various types of carbonaceous raw materials like coal, pine and eucalyptus wood, coconut shell, rice husk, saw dust, lignite, etc.

3.7.3 Adsorbent Mass Requirement

The amount of air impurity which is adsorbed in adsorber columns is the amount that remains in the helium after condensation in tube-in-tube heat exchanger and subcooler. After liquid air separation, impure helium is saturated with air at 77 K which will be adsorbed by activated charcoal in adsorber columns.

Two cases are analysed for calculating the amount of impurity concentration remaining in helium stream after liquid air separator vessel. The volumetric flow rate of helium through purifier is 20 nm^3/hr at a minimum adsorption pressure of 120 bar(a).

(1) Impure helium containing 40% air impurity

(2) Impure helium containing 40% nitrogen impurity

1) 40% Air Impurity in Helium

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) 40% Nitrogen Impurity in Helium

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 nm^3/hr for 6 hr purification process.

Quantity of nitrogen to be adsorbed

= 0.83% of 20 nm^3/hr flow rate for 6 hr

= [(0.83 x 20 x 6) / 100] nm^3

= 0.996 nm^3

= 0.996 x 10^6 cc

181.5 cc/gm is the volume of nitrogen needed to form a monomolecular layer of gas over the total adsorbent surface (for a certain type of activated charcoal) per unit mass of adsorbent when temperature is 77 K as mentioned in gas adsorption parameters for BET relationship [2].

Hence, activated charcoal requirement

= (0.996 x 10^6) / 181.50

= 5.487 kg

This helium purifier works on thermal regeneration, so estimation of adsorbent requirement is based on 70% saturation of adsorber columns as suggested by Haselden [6].

Hence, quantity of activated charcoal requirement

= 5.487 / 0.70

= 7.84 kg

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

Remaining Nitrogen Impurity at 150 bar(a)

The following calculation shows the remaining nitrogen impurity in helium stream after liquid air separator vessel at 150 bar(a) delivery pressure.

Input parameters are:

Impure helium has 40% nitrogen impurity

Saturation pressure of nitrogen at 77 K is 1 bar

Partial pressure of nitrogen at 77 K = 1 bar

Purifier delivery pressure and flow rate= 150 bar(a) and 20 nm^3/hr

Mole Fraction of nitrogen at 77 K after liquid air separator

= (1 / 150) x 100

= 0.67%

Helium containing 0.67% remaining nitrogen enters the adsorber columns after liquid air separator vessel.

3.7.4 Design of Cross-section of Adsorber Column

High process flow velocity through the adsorber columns will cause bed fluidization, powdering of charcoal particles and lack of sufficient time for proper

adsorption on adsorbent surface. Therefore, helium stream velocity should have optimum value which is calculated hereafter.

Superficial velocity of helium through fixed adsorber column ranges approximately between 10^{-2} to 1 m/s [7]. The dimensions of adsorber columns have been designed to keep gas velocity near 10^{-2} m/s or lower.

Delivery pressure of purifier = 150 bar(a)

Volumetric flow rate of helium = 20 nm^3/hr at 300 K ambient

Therefore, flow rate of helium at 150 bar(a) and 77 K = 9.44 x 10^{-6} m^3/s

Flow rate of helium at 150 bar(a) and 77 K = (π/4) x $(D_i)^2$ x V

Where,

D_i = Internal diameter of adsorber column

V = Superficial velocity = 10^{-2} m/s

Using above equation,

9.44 x 10^{-6} = (π/4) $(D_i)^2$ x (10^{-2})

Or, D_i = 0.03467 m = 34.67 mm

Inside diameter of adsorber column comes out to be 34.67 mm. Such pipe should be selected whose inside diameter is 34.67 mm or higher. The adsorber columns are immersed in LN_2 and also exposed to 120°C in a cyclic manner, so austenitic stainless steel of specification ASTM A 312 TP 316L has been chosen as adsorption column pipe material.

Let us consider two options for pipe size selection:

1) 40 NB
2) 50 NB

The allowable internal pressure is calculated by using the following formula [9]:

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 SS 316L

Minimum wall thickness, t_{min}

Nominal outside diameter, OD

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

Y= {OD - (2 x t_{min})} / 2(OD – t_{min}) for $t_{min} = OD/6$

Thickness Calculation with Pipe Size of 40 NB

Outside diameter of 40 NB pipe is 48.3 mm

Applying Eqn. (i),

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

Or, $t_{min} = 3.03$ mm

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

Thickness Calculation with Pipe Size 50 NB

Outside diameter of 50 NB pipe is 60.30 mm

Applying Eqn. (i),

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

Or, $t_{min} = 3.78$ mm

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

From the above two calculations, it is clear that both 50 NB Sch 40 and 40 NB Sch 40 can be chosen. If 40 NB Sch 40 is chosen, number of adsorber columns is 7 [refer subsection 3.7.5 for analysis] which needs bigger LN_2 vessel resulting in higher LN_2 consumption. Therefore, 50 NB Sch 40 is chosen by optimising the dimension and layout of purifier components within LN_2 vessel. Sudden pressure and temperature surge during purifier operation demands higher value of thickness. To be safe, 50 NB Sch 80 pipe has been chosen.

Superficial velocity of helium in 50 NB Sch 80 (ID = 0.04922 m) pipe

$= (9.44 \times 10^{-6}) / [(\pi/4) (0.04922)^2]$

$= 4.96 \times 10^{-3}$ m/s.

Velocity through Charcoal Packed Adsorber Columns

Internal volume of 5 adsorber columns = 0.013 m^3 [refer section 3.9]

Total length of 5 adsorber columns = 10 m [refer subsection 3.7.5]

Let 'd_e' m diameter of a pipe having length and volume 10 m and 0.013 m^3 respectively.

Equating,

$0.013 = \pi/4 \times d_e^2 \times 10$

Or, $d_e = 0.04$ m

Actual velocity of helium through packed charcoal columns

$= [(9.44 \times 10^{-6}) / (\pi/4 \times 0.04^2)]$ m/s

$= 7.5 \times 10^{-3}$ m/s

This velocity is favourable for efficient adsorption.

3.7.5 Adsorber Column Length Calculation

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

= 550 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 3.7.3]

Therefore,

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

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

= 7.49 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

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

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

= 0.003805 m^3

Charcoal content of each column = 0.003805 x 550 = 2.093 kg

Therefore, total amount of charcoal accommodated in 5 adsorber columns

= 2.093 x 5

= 10.5 kg

3.7.6 Pressure Drop Calculation

Determination of pressure drop of helium flow in 5 adsorber columns is based on Ergun equation. 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 adsorber columns,

$f_p = (150 / Re) + 1.75$

$f_p = (\Delta p / L) [D_p / (\rho \times V_s^2)] [\epsilon^3 / (1 - \epsilon)]$

Where,

Reynold's Number, $Re = (D_p \times V_s \times \rho) / \{(1 - \epsilon) \mu\}$

Equivalent spherical diameter of particle, D_p

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

= 0.003 m

Superficial velocity, V_s

= (Volumetric flow rate / Cross sectional area of column)

The basic input parameters for design are as follows:

Delivery pressure of purifier = 150 bar(a)

Operating temperature = 77 K

Density of helium at 150 bar(a) and 77 K, ρ = 70 kg/m^3

Specific heat of helium, C_p = 5.4 kJ/kg-K

Dynamic viscosity of helium at 150 bar(a) and 77 K, $\mu = 51.5 \times 10^{-6}$ Pa.s

Flow rate of helium = 20 nm^3/hr

Particle diameter of charcoal granules = 3 mm (average value)

Bulk density of charcoal = 550 kg/m^3

Void fraction or porosity, ϵ = 0.6 [2]

Length of 5 adsorber columns, L = 10 m

Adsorber column size is 50 NB Sch 80 (ID = 0.04922 m)

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

= 0.034 m^3/hr

= 9.44×10^{-6} m^3/s

V_s = $(9.44 \times 10^{-6}) / [(\pi/4) \times (0.04922)^2]$

= 4.96×10^{-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

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

Δp = 50 Pa

= 50×10^{-5} bar

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

Table 3.3: 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	50×10^{-5} bar

3.8 Design of LN_2 Vessel of Purifier

LN_2 vessel has been designed as per ASME Section VIII Division I. Material of construction chosen for inner and outer vessel, bottom and top flanges is SA 240 TP 304L.

The vessel accommodates five adsorber columns, six tubular heaters, two heat exchangers, one snow filter, one LN_2 level indicator, one Liquid Air Separator Vessel, connecting tubes and a cage. Optimising the location and size of all components within

LN_2 vessel, inside diameter and depth of inner vessel comes out to be 600 mm and 1600 mm respectively.

3.8.1 Analysis of LN_2 Consumption

LN_2 is required to cool the adsorber columns and two heat exchangers to 77 K. The exact values of LN_2 requirement for cool down and purification phase can be obtained only with experiment. An analysis of total heat leak of the mechanical system and estimation of LN_2 requirement during purifier run for 6 hr is done considering the actual operating conditions which are illustrated hereunder.

During operation, heat ingress within LN_2 chamber takes place due to the following reasons:

- Heat in-leak from ambient to LN_2 chamber through superinsulation i.e. static evaporation loss
- Heat evolution due to adsorption process
- Cooling down of impure helium gas to 77 K and condensation of impurities
- Heat conducted through the tubes connected to top flange of LN_2 vessel.

Estimation of Static LN_2 Evaporation Loss of LN_2 vessel

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

Inner and outer vessel shell wall thickness = 4 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

Cp of helium = 5.20 kJ/kg-K

Cp 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]

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

Capacity of empty LN_2 vessel

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

$= 388\ l$

The following calculations take into account the average LN_2 level of 1350 mm in LN_2 vessel i.e. average LN_2 level is 0.25 m from 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 hereunder:

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

$= 14.13$ l of LN_2 evaporation / 6 hr

Therefore, static LN_2 evaporation loss for 6 hr is 14.13 l

Heat Evolved due to Nitrogen Adsorption

Helium gas entering adsorber columns carries 0.83% remaining nitrogen impurity at the flow rate 20 nm^3/hr for 6 hours.

Amount of nitrogen in helium flow per hr

= (20 x 0.83) /100

= 0.166 m^3 of N_2

= [(0.166 x 1000) / 22.4]

= 7.40 mole of N_2 in helium flow per hour

Characteristic energy of adsorption of N_2 on charcoal, E

= (2940 ± 70) J/mole [19],

Maximum value 3010 J/mole have been adopted for analysis

Energy of adsorption released in 6 hr operation

= (7.40 x 3010 x 6) J

= 133.64 kJ

Hence, LN_2 boil-off in 6 hr due to release of energy of adsorption

= [(133.64 x 1000) / (199.30 x 813.89)] l

= 0.83 l

LN_2 Requirement for Condensing Nitrogen Impurity

Amount of nitrogen impurity condensed in 6 hr

= (40 − 0.83)% of helium flow rate

= 20 x (40 − 0.83)% x 6 x 1.124 kg

= 52.8 kg

LN_2 consumed to condense 52.8 kg nitrogen impurity

= (52.8 x 1000 / 813.89) l

= 64.9 l

Total LN_2 Consumption for 6 hr Purification Run

LN_2 consumed in 6 hr purification run

= (Static evaporation loss + LN_2 consumed due to adsorption heat evolution + LN_2 boil off for nitrogen impurity condensation)

= (14.13 + 0.83 + 64.9) l

= 79.86 l

3.9 Time Estimation prior to Cylinder Filling

In the purification phase, after start up compressor takes certain time to fill the purifier to 120 bar(a) at a flow rate of 20 nm^3/hr. Back pressure regulator permits the gas to flow downstream once purifier reaches the minimum adsorption pressure of 120 bar(a). For estimating the time required to fill the purifier, internal volumes of the

purifier components located before back pressure regulator have been calculated which are shown in Table 3.4.

Table 3.4: Internal volumes of components

Component	**Materials for fabrication**	**Internal volume** (m^3)
Moisture separator vessel	50 NB Sch 80 pipe, End caps	7×10^{-4}
Shell and tube heat exchanger	50 NB Sch 80 pipe, end caps, 6 mm OD tube	4.9×10^{-4}
Tube-in-tube heat exchanger	16 mm and 10 mm OD tube	1.253×10^{-3}
Subcooler	10 mm OD tube	6.03×10^{-4}
Liquid air separator vessel	100 NB Sch 80 pipe, end caps	4.75×10^{-3}
Snow filter	50 NB Sch 80 pipe, end caps, wire cloth	1.9×10^{-4}
Tubes, fittings and valves	Tube OD 6 and 10 mm, Valves and fittings for 6 mm OD tube	4.5×10^{-4}
Five Adsorber Columns	50 NB Sch 80 pipe, end caps, U-bend, End filter, charcoal, 6 mm socket	0.013 (calculation shown below)
Total		0.0215

The result shows that helium gas occupies volume of 0.0215 m^3 at 120 bar(a) and 77 K in purifier which is equivalent to 10.1 nm^3 at ambient temperature i.e. 300 K. Time required to raise the pressure of purifier to 120 bar(a) after compressor start up

= (10.1 / 20) hr

= 30 min

<u>Estimation of Internal Volume of 5 Adsorber Columns</u>

Internal volume of pipe of 10 m length = (π/4 x ID^2) x total length

= 0.019 m^3

Internal volume of 5 U-bends = 0.002264 m^3

Internal volume of 10 end caps = 3.27×10^{-4} m^3

Volume of pipes, U-bends, end caps = ($0.019 + 0.002264 + 3.27 \times 10^{-4}$) m^3

= 0.021 m^3

Volume occupied by 10 filter units at inlet and outlet of adsorber column = 1.7×10^{-4} m^3

Quantity of activated charcoal used is 10.5 kg

Density of charcoal = 550 kg/m^3 [refer Table 3.1]

Porosity of activated charcoal = 60% [22]

Volume of activated charcoal = 10.5 / 550 = 0.019 m^3

Void space within charcoal = (0.019 x 0.6) = 0.0114 m^3

So, Volume of 5 adsorber columns which is occupied by gas

= (Volume of pipes, U-bends, end caps – Volume of filter units – Volume occupied by activated charcoal + Void space within charcoal)

= 0.013 m^3

3.10 Selection of Regeneration Process

Regeneration procedure of adsorbent columns is of two general types:

- The Pressure Swing Cycle
- 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^oC 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 for Regeneration

The adsorber columns are heated by six tubular heaters positioned between adsorber columns within the LN_2 vessel. The heater specification chosen is 1 kW, 220 VAC, single phase, 50 Hz. Tubular heaters have been arranged in such a way 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, 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

The estimated 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.

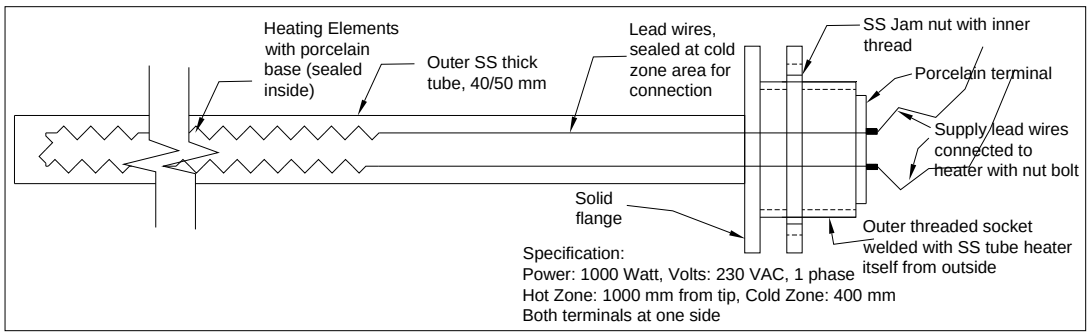

Fig. 3.8: Schematic diagram of a tubular heater

Chapter 4

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 $-196^{o}C$ to $120^{o}C$ 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 foolproof 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.

4.1 Fabrication of Moisture Collector and Liquid Air Separator Vessels

In the moisture collector vessel, condensed moisture and oil gets separated from compressed helium gas and helium escapes through the top of this unit at ambient temperature. In liquid air separator vessel, helium and condensed liquid air enter at 77 K where they are separated by cyclonic movement of gas. The gaseous helium escapes through top of the vessel while the condensate falls to the bottom.

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) Pipe 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 cap, 50 NB Sch 80, seamless with butt weld ends, dimensions conform to ANSI B 16.9, ASTM A312 TP 316L
(4) End cap, 100 NB Sch 80, seamless with butt weld ends, dimensions conform to ANSI B 16.9, ASTM A312 TP 316L
(5) Tube, 6 mm OD, thickness 1 mm, SS 316L
(6) Tube, 10 mm OD, thickness 1 mm, SS 316L
(7) Socket fittings for the tubes

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. The inserted tubes were welded directly on the pipe surface. All the other ports have sockets welded to vessel body. Fig. 4.1 shows the liquid air separator vessel under fabrication; here 10 mm OD tube is tangentially welded to the pipe and one end is closed with end cap.

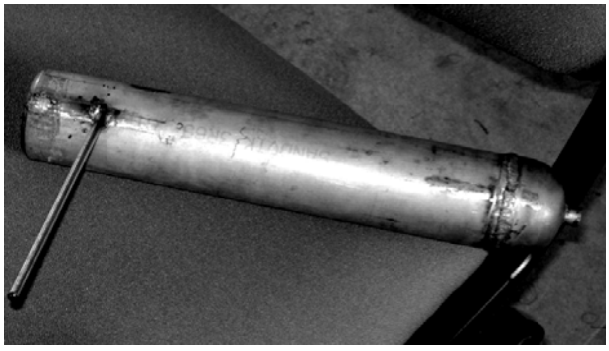

Fig. 4.1: Liquid air separator vessel under fabrication

4.2 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, dimensions conform to ANSI B 16.9, ASTM A312 TP 316L
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. Bottom end cap has two ports while the top cap has one port at the centre. Top port is coil inlet port and outlet is the bottom off-centre port. Drain line is the centre port of the bottom cap. Vessel was welded to 6 mm OD tube through sockets. Radiography was done to butt welds after fabrication. This heat exchanger was insulated with fibre glass insulation. Fig. 4.2 shows the bottom part of the heat exchanger under fabrication.

Fig. 4.2: Shell and tube heat exchanger before closing one end

4.3 Fabrication of Tube-in-Tube Heat Exchanger

Tube-in-tube heat exchanger, located in the ullage space i.e. in the vapour region of LN_2 vessel, cools down the impure helium stream to approximately LN_2 temperature by the return purified helium stream. This 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. This concentric counterflow design produces

turbulent conditions at low flow rates, increasing heat transfer coefficient and hence the rate of heat transfer.

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. A special fixture was fabricated for coiling manually. Helical coiling was done manually followed by welding of end fittings. The diameter of the coil was made 490 mm to match the cage dimensions [refer section 4.10 for description of cage]. 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.4.3: Tube-in-tube heat exchanger

4.4 Fabrication of Subcooler Heat Exchanger

Subcooler heat exchanger, situated after tube-in-tube heat exchanger, is submerged in liquid nitrogen. It 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.

This type of heat exchanger is the most efficient, compact, lightweight, cost effective and easy to fabricate, and is suitable for high pressure and low flow

applications. 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.

4.5 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.

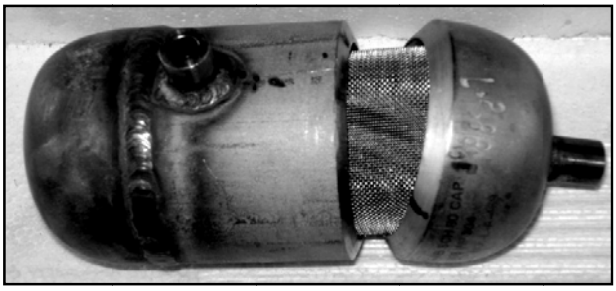

Fig. 4.4: Snow filter before final welding

4.6 Fabrication of Adsorber Columns

The adsorber columns, containing activated charcoal, remove remaining air contaminants from impure helium flow that have not been condensed out in the heat exchangers, and thus yield Grade 4.5 helium. 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, dimensions conform to ANSI B 16.9, ASTM A312 TP 316L
3) End cap, 50 NB Sch 80, forged, seamless with butt weld ends, dimensions conform to ANSI B 16.9, ASTM A312 TP316L

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 air gases. Specification: Type 5A, bead form, 4 mm diameter.

The fabrication procedure explained here is for one adsorber column. Same procedure has been followed for fabricating all other columns.

Column

For the construction of an adsorber column, 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. The socket was welded to the U-bend body and then the plug was welded to the socket. 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. Also, sockets were welded with the end caps of adsorber column. The top end socket diagram is shown in Fig. 4.8. Fig. 4.5 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. 4.7. 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.

Conditioning of Charcoal

Activated charcoal obtained from market was filtered with proper sized wire screens to collect uniform sized (– 4 + 8 BSS) grains. After gathering these grains, they were cleaned with compressed air to remove charcoal dust as much as possible. It is

better to regenerate the charcoal in laboratory oven at 150^{o}C, before filling in adsorber columns. In the absence of an oven in the laboratory, we did the regeneration of charcoal during conditioning of purifier after assembly, which is detailed later.

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.

Welding of the Elements

Two filter units were welded at both the ends of adsorber column by taking proper precaution, so that welding heat would not be conducted to charcoal. Welding was done at 25 mm depth from the ends of the column. Fig. 4.6 shows a filter unit welded to the pipe of adsorber column. Lastly both the ends were closed by welding end caps.

Before welding, edge preparation for butt weld was done by grinding. Three runs were given for butt welds; first run was done by 1.60 mm diameter welding rod and other two runs by 2.60 mm welding rod. Radiographic examination was done on all the butt joints viz. U-bend with pipe piece, end cap with pipe piece, and DP (Dye Penetration) test was carried out on the remaining joints i.e. filter housing with pipe, socket with end caps, etc. During TIG welding, argon flow was 10 l/min for purging and 5 l/min for welding. Root gap provided was 2.50 to 3 mm. Charcoal filled columns were then purged with high pressure dry nitrogen from both ends to remove charcoal dust. Height of adsorber column comes out to be 1144.40 mm.

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. 4.10.

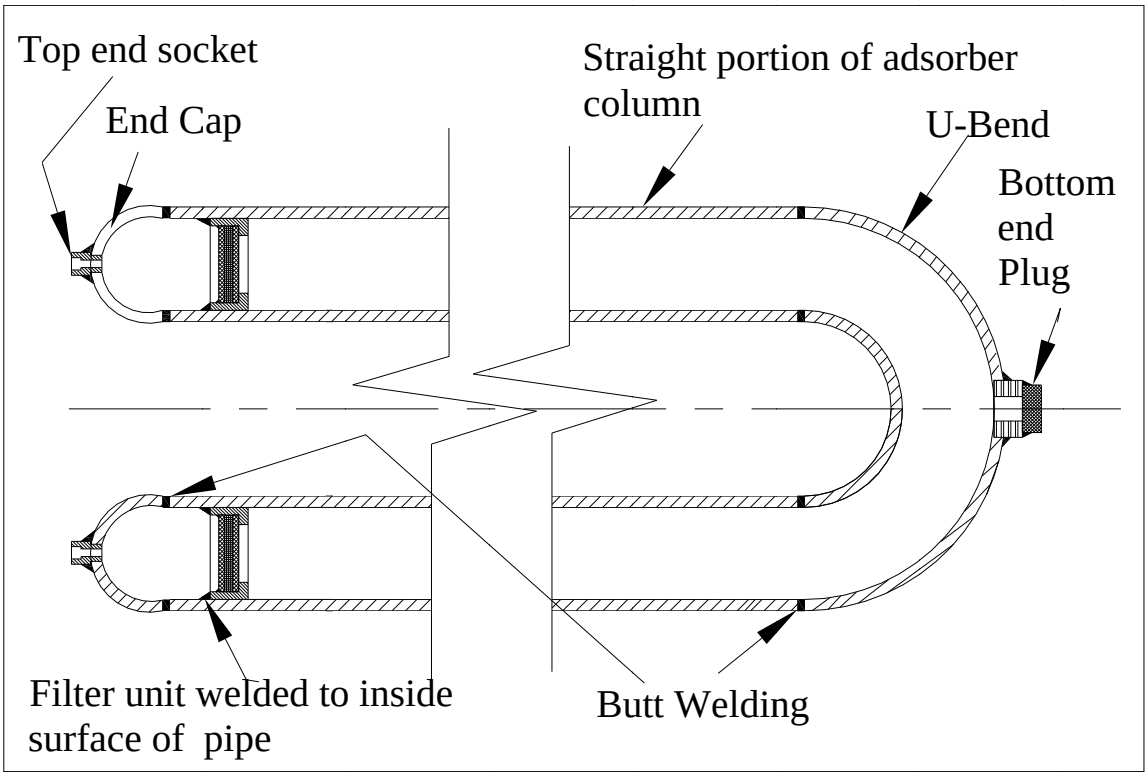

Fig. 4.5: Sectional view of the adsorber column

Fig. 4.6: Filter unit welded at one end of adsorber column

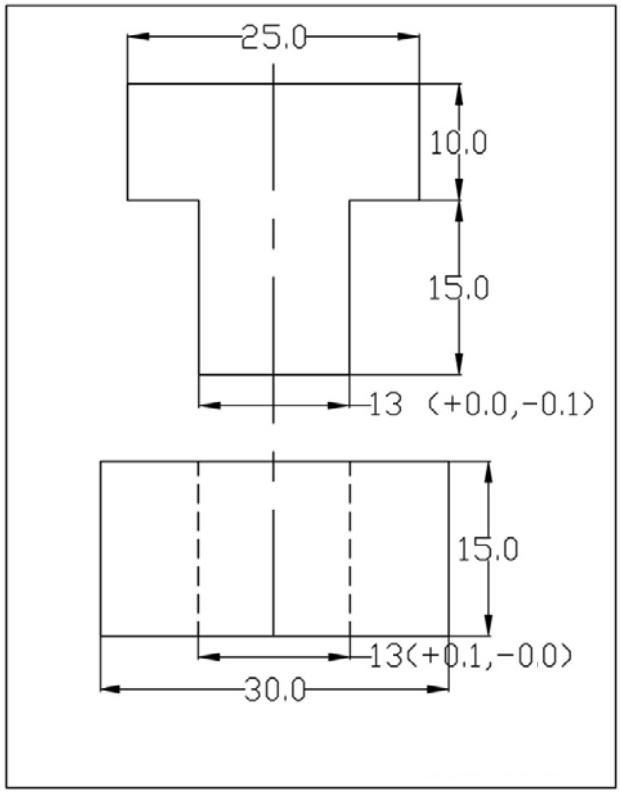

Fig. 4.9: Bottom end plug and socket of adsorber column

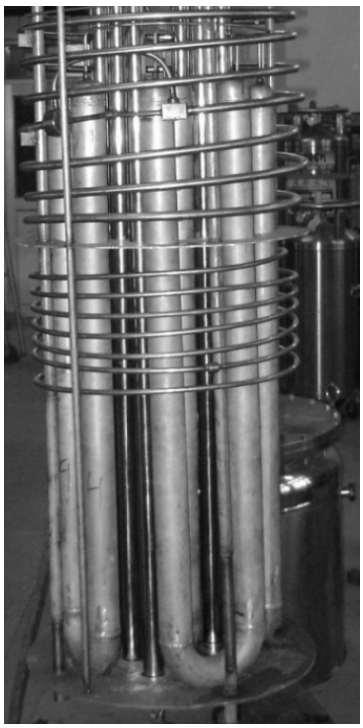

Fig. 4.10: Adsorber columns in assembled condition

4.7 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. A control panel containing all valves and gauges was placed at convenient location.

4.8 Fabrication of LN_2 Vessel of Purifier

The applicable code for the design and fabrication of LN_2 vessel is ASME Section VIII Div I. LN_2 vessel is double walled superinsulated vessel which consists of two parts:

1) Top flange
2) Vessel, which is superinsulated

The vessel was fabricated by M/s Srinitech Services, Mumbai. The specification of the fabricated LN_2 vessel is shown in Table 4.1.

Fig. 4.11 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. Short pieces of tubes of 100 mm lengths were welded to all ports on both the sides of top flange for convenience in welding during assembly. LN_2 vessel drawing in Appendix II shows the dimensions.

Table 4.1: Specification of LN_2 vessel

Sl.	Parameter	Value
1.	Inner Vessel Outside diameter Depth Shell thickness Dished end thickness Material of construction	 600 mm 1600 mm 4 mm 5 mm SA 240 TP 304L
2.	Outer Vessel Outside diameter Shell thickness Dished end thickness Material of construction	 700 mm 4 mm 5 mm SA 240 TP 304L
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.	Annular space Gap Vacuum Adsorbent used	 46 mm 10^{-6} mbar Molecular sieve
6.	Helium leak tightness of welds	1×10^{-9} mbar-l/s
7.	Evacuation port size	ISO KF 25

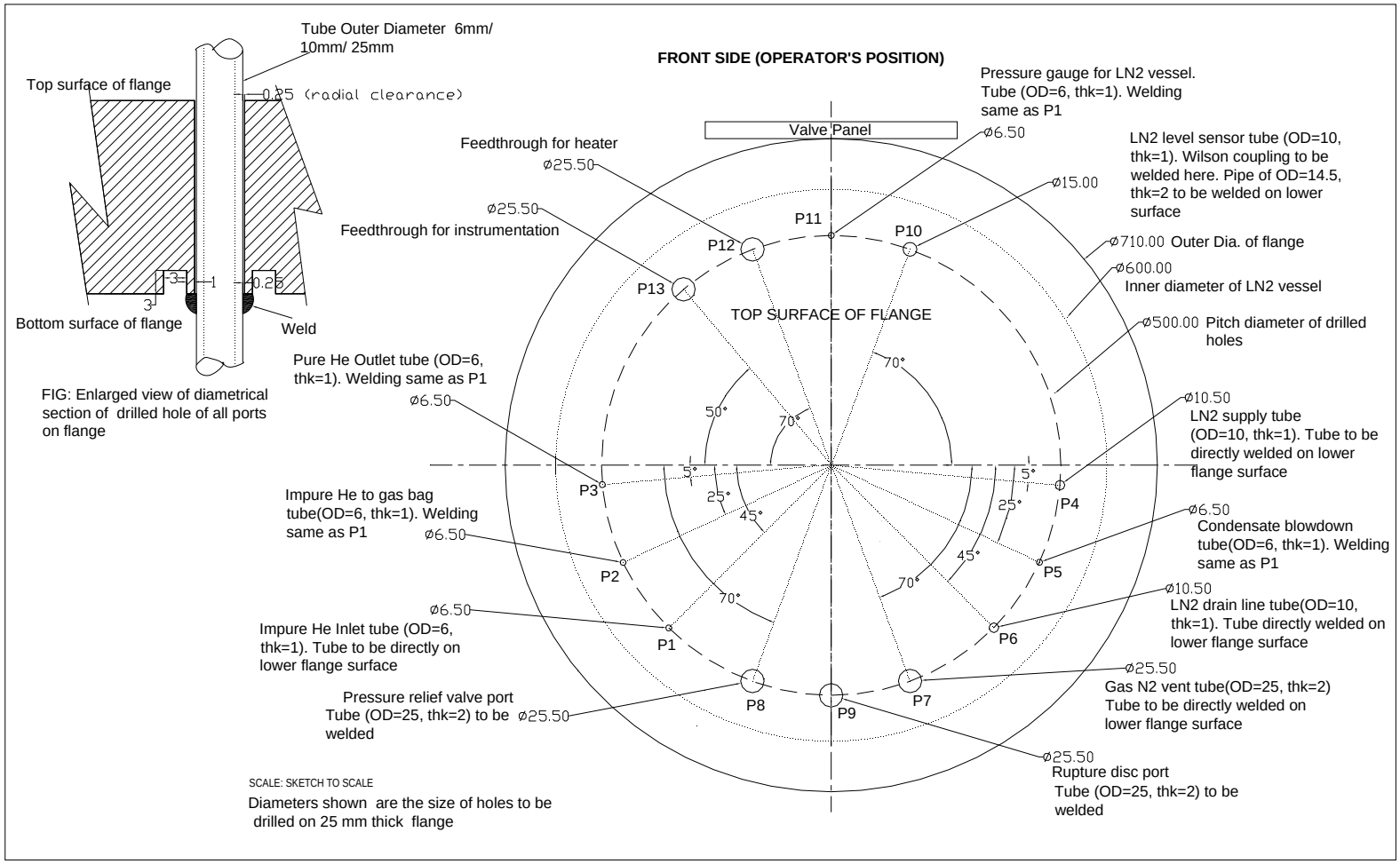

Fig. 4.11: Location of ports on the top flange of LN_2 vessel

4.9 Fabrication of Tubing Connections for Helium Gas and LN_2

The tubes used in this work for LN_2 and helium gas transmission are austenitic stainless steel grade 316L conforming to ASTM standard.

The brief specification [3] of the tubes purchased for helium purifier is mentioned below.

- Material: Seamless tube material as per ASTM A 269 TP 316L
- Surface finish: Bright annealed finish (BA or 2BA) for tubes. A finish that is designated "2BA" has been bright annealed and then passed between highly polished rolls.
- Tube outer diameter tolerance: As per ASTM A269
- Hardness: Max HRB 80
- Tolerance on wall thickness: $\pm$ 10%

The various sizes of the tubes used in this work are mentioned in Table 4.2.

Table 4.2: Tube sizes used in purifier

Sl	Item description of tube Outside diameter, wall thickness,(mm, mm)	Material
1.	3.18, 0.71	316L
2.	6, 1	316L
3.	10, 1	316L
4.	16, 1.5	316L
5.	25, 2	316L

The most often used joints encountered in stainless steel instrumentation tubing for helium are TIG welding joint and the demountable joint i.e. compression type ferrule fitting. In helium purifier system, the tubing at ambient temperature have ferrule type joints for connecting valves and gauges. Fig. 4.11 displays all the ports on top flange of helium purifier through which all high pressure tubes connecting ambient temperature components with the cryogenic temperature components. The figure also shows the clearances provided for welding tube with thick top flange. The joints which are exposed to cryogenic temperature are TIG welded [8] which ensures leak free joints at high pressure and LN_2 temperature.

Helium users globally use ferrule connectors of some specific brands like Swagelok, USA & M/s Parker, USA. M/s Swagelok products have been adopted in this

work as their product has global acceptance for their robustness, easy operation, excellent helium leak tight sealing even in stressful and harsh environments.

The LN_2 supply line was fabricated out of 10 mm OD tube. LN_2 vessel is pressurized to drain LN_2 to external dewar. Bottom end of drain line almost touches the bottom of LN_2 vessel to recover the maximum possible LN_2. Drain and vent line were fabricated from 10 mm and 16 mm OD tube respectively.

Wilson Coupling

Wilson coupling is a demountable type of coupling, generally used for cryogenic purpose. The LN_2 level sensor which was inserted in LN_2 from top is clamped by this coupling. This coupling was welded on the top flange of LN_2 vessel. It consists of stainless steel body, two neoprene O-rings, two washers and a nut to hold level sensor tube. Two O-rings were provided for ensuring better leak tightness. This leak proof coupling helps to maintain pressure within the LN_2 vessel.

Valves

The different types of the valves used in the system are ball valve, needle valve, pressure safety relief valve, pressure reducing as well as back pressure regulator and non return valve. There are a total of 23 valves used in purifier. All the valves used were connected with removal type connections so that any defective valve could be replaced. Ball valves and needle valves were selected based on pressure rating, flow rating and temperature rating. A solenoid valve suitable for cryogenic purpose was used in LN_2 supply line which was controlled as per LN_2 level controller. Non return valve was used in the boil-off LN_2 vent line to prevent air and moisture entry inside purifier vessel.

Safety relief valves, SRV1 and SRV2 [refer Fig. 3.1 for nomenclature] were provided before and after the charcoal columns to take care any pressure build up of helium stream. SRV3 is for pressure relief of purifier LN_2 vessel. Rupture disk was provided in LN_2 vessel for backing up the safety relief valve. If relief valve fails to operate or cannot relieve enough pressure fast enough, the burst disk will rupture and release the overpressure. Safety relief valve is set at a pressure of 0.5 bar(g). In rupture disk, burst pressure is 1 bar(g) at burst temperature of 22^{o}C.

Needle valves were provided before pressure gauges to replace defective gauges while the system in operation. Inline micron filters of 7 micron were used here at inlet of charcoal columns for particulate matter filtration and at outlet for charcoal dust filtration from helium stream. All the valves except the valves used in LN_2 supply line, drain line

and vent line, were placed on valve panel. Valve panel of dimension 710 mm x 600 mm was fabricated from 3 mm thick SS 304 plate. The panel was supported by 25 x 25 SS 304 square tubes and this panel was placed in front of the purifier vessel. Brief specification of the valves and micron filters selected for helium purifier are mentioned in Appendix I.

4.10 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, as shown in Fig. A2, A3 and A4 in appendix II. 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.

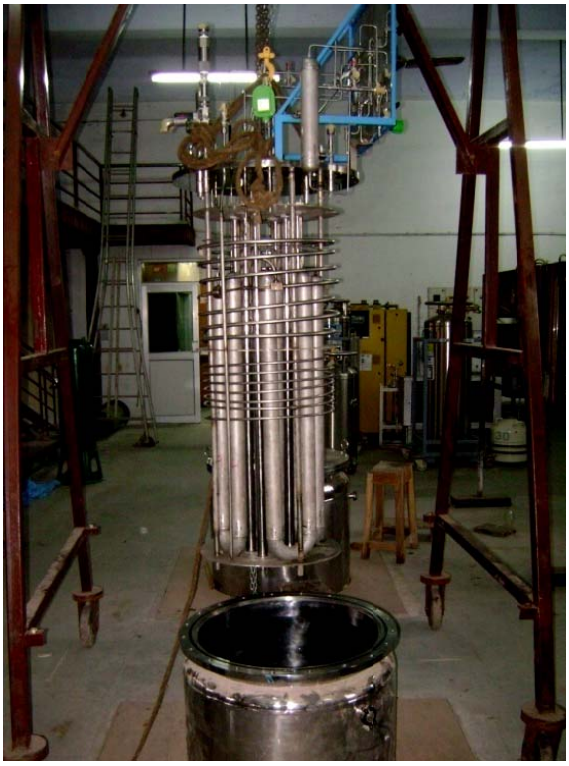

Fig. 4.12: Assembly process of helium purifier

4.11 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 completion of welding of adsorber columns, D1 and D2, circumferential butt welds were 100% examined with X-ray source in accordance with ASME Section V. All the joints passed the test. Dye penetration test was carried out where radiography was not possible i.e. the socket joints, fillet joints, corner joints, etc.

Pneumatic Leak Test

After the 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. After completion of test, the system pressure was gradually vented out.

4.12 Conditioning of Helium Purifier

After the leak test, the purifier was heated by tubular heaters and evacuated by rotary vacuum pump up to 10^{-3} mbar for a few days. The system was purged with Grade 4.5 helium and evacuated. 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.

4.13 Safety Features

The system operates at low temperature and high pressure, so several protective devices are provided in the compressor, LN_2 vessel and other components of purifier. LN_2 vessel is provided with a safety relief valve as well as a rupture disk. Safety relief valve is set at a pressure of 0.5 bar(g). In rupture disk, burst pressure is 1 bar(g) at burst temperature of 22^{o}C. Nitrogen gas vent line of the vessel is directed outside the laboratory.

High pressure helium gas lines are protected against overpressure by two safety relief valves, SRV1 and SRV2 [for nomenclatures refer Fig.3.1]. The set pressure of the SRV's are 165 bar(g) [9]. During regeneration, adsorber columns are protected against excessive pressure by SRV2.

Compressor is fitted with the safety devices like high and low pressure safety switch, safety relief valves, overload protection relay, etc.

Gas bag works at low pressure of 50 mbar at ambient temperature, so an oil pressure based safety relief valve is provided with gas bag system. The oil level determines relief pressure of the system.

Helium and nitrogen are chemically inert. Sudden spillage of LN_2 or leakage of helium gas will reduce the oxygen level in air which may cause asphyxiation to the persons working in confined laboratory. Installation of an oxygen monitor with both audible and visible alarms has been planned.

4.14 Vendor Development

For the development of purifier system, utmost efforts were made to purchase most of the components from the indigenous manufacturers and thereby giving boost to Indian industries to work with cryogenic technology. But, only a few items which were not at all possible to manufacture indigenously, were imported. The vendors and manufacturers list of various components is provided below in tabular form.

Table: 4.3: List of suppliers and manufacturers of components

Sl.	Item's name	Supplier / Manufacturer
1	Helium Gas Bag, Cap. 5 m^3	M/s Softex Industrial Products Pvt. Ltd, Kolkata
2	Reciprocating Compressor	M/s Metec Corporation, South Korea
3.	Activated charcoal	M/s Exal Corporation, Vadodara
4.	Molecular seive	M/s Pharma-Chem-De-Drugs, Kolkata
5.	LN_2 vessel of purifier	M/s Srinitech Services, Mumbai
6.	Pipes and Tubes	M/s Sandvik, Sweden
7.	Tubular heater	M/s Canteen Equipment Trading Co., Kolkata
8.	Ball valve, Needle Valve, Check Valve, NRV, micron filter, SRV	M/s Swagelok, USA

Sl.	Item's name	Supplier / Manufacturer
9.	Back Pressure Regulator, Pressure Reducing Regulator	M/s Swagelok, USA
10.	Ferrule fittings	M/s Swagelok, USA
11.	Cryogenic Safety relief valve	M/s Herose, Germany
12.	Burst disk	M/s BS&B Safety Systems (India) Ltd., Chennai
13.	Cryogenic Solenoid Valve	M/s Asco (India) Ltd, Chennai
14.	Pressure gauges	M/s Wika, Pune, M/s Swagelok, USA
15.	Pt100 RTD	M/s D B Products and Systems, Kolkata
16.	U-Bend, Cap	M/s Arvind Pipes & Fittings Industries Pvt Ltd, Mumbai
17.	Sample Cylinder	M/s Swagelok, USA
18.	Multi component detector	M/s Linde, Germany
19.	Cylinder	M/s Everest Canto Cylinder Ltd., Mumbai
20.	Cylinder Manifolds	M/s Cryogenic Concern Pvt. Ltd., Kolkata
21.	Purifier Fabrication	M/s Cryogenic Concern Pvt. Ltd., Kolkata

Chapter 5

OPERATION AND RESULTS

This chapter illustrates the experimental set up and operational procedure followed in the first run of the helium purifier. Operation process was performed after final installation and commissioning of helium purifier. Purifier was operated in three steps:

1) Cooling down of purifier
2) Purification
3) Regeneration

In regeneration phase, the operator ensured the complete regeneration of charcoal adsorbent, and drying of three heat exchangers and connecting tubing. After regeneration procedure, the purifier system was ready for next run.

5.1 Operation of Purifier

The operation of the purifier was conducted once for one session at NIT, Rourkela. For this experiment, Grade 4.5 helium was purchased from M/s Praxair India Private Limited, Thane, Maharashtra, in cylinders. 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 gas was supplied from one manifold and nitrogen from cylinder by passing through rotameter at the flow rate of 38 l/min for helium and 2 l/min for nitrogen. Impure helium prepared in gas bag consisted of 5% nitrogen and 95% helium. This impure gas was compressed by compressor to deliver the pressure of 150 bar at flow rate of 20 nm^3/hr for purification. The purified gas from purifier was collected in empty cylinders in manifold and samples were collected in sample cylinders.

Modes of Operation

Helium purifier operation procedure consists of sequentially cycled steps. They are as follows:

Cool Down Phase

LN_2 vessel of purifier was filled from external portable LN_2 dewar at pressure of 0.2 bar(g) by using the pressurization system of dewar. In the first run, around 300 l of LN_2 was used to cool down and fill the LN_2 vessel. LN_2 level in the vessel was 1300 mm which was measured by stainless steel dip stick.

Purification Phase

This was the purification stage for purifying impure helium by operating compressor. Compressor delivered compressed helium to the purifier at a flow rate of 20 nm^3/hr. Firstly, valve BV6 [refer Fig. 3.1 for nomenclature] was opened and compressor was switched on. Back pressure regulator allowed helium to flow out of system once the purifier pressure reached the minimum adsorption pressure of 120 bar(a). 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 150 bar. Fig.5.1 shows the photograph of ongoing purification process.

Four samples were collected in Swagelok make sample cylinders (water capacity 75 cc) from outlet line of purifier at time intervals, which were sent to VECC, Kolkata, for analysis. Before sample collection, SS 304L sample cylinders were baked, evacuated and purged with Grade 4.5 helium to ensure contaminant-free inner atmosphere.

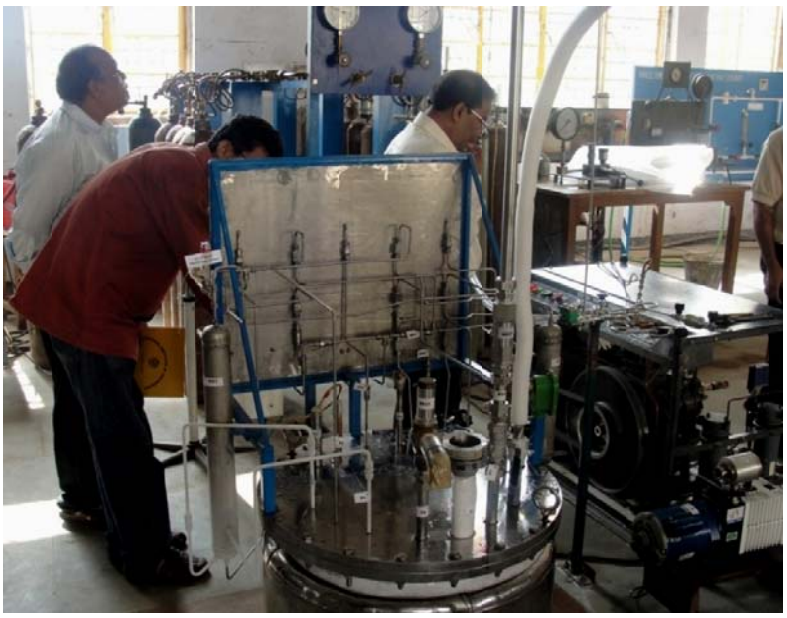

Fig. 5.1: Operation of helium purifier

Regeneration Phase

After purification was over after one session of operation, LN_2 vessel was decanted by pressurization. Regeneration of the purifier was carried out by means of heating and evacuation. The system was backfilled with Grade 4.5 helium. The system was ready for the next run beginning with "cool down" phase.

5.2 Results of the Experiment

The first sample of the purified helium was collected 45 min after the purifier began cylinder filling. The second, third and fourth sampling were done at an interval of 30 min. 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 5.1: 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 6

CONCLUSION

The final chapter of the thesis literarily refers to the ultimate episode of this MTech (Research) dissertation. Beyond the literary meaning, this chapter characterizes the first footprint of the major strides to be taken up in future. Let us try to figure out the achievements and failures of our primary endeavor followed by the next courses of action to be adopted.

Concluding Remarks

This work is a tiny contribution in the larger world of cryogenic technology. Indigenous development of helium purifier shall not only lead to economy for the researchers and academicians of India but shall give a quantum jump in cryotechnology in our nation. Summarizing, the following may be seen as the significant contributions of the present investigation.

- ✓ The dissertation presents an updated literature review on almost all aspects of cryogenic adsorber based helium purifier and may serve as a ready reference for future work.
- ✓ This thesis presents the design of adsorber columns and other components of purifier and selection of low cost helium compressor
- ✓ Coconut shell activated charcoal columns have worked fine in the first run, but we need to observe its performance in long run if purifier is operated regularly for one year.
- ✓ Vendors and manufacturers have been identified in India for fabricating different components of cryogenic equipment
- ✓ A gas bag for storing helium at low pressure has been developed indigenously.
- ✓ The helium purifier has been successfully commissioned.

Scope for Further Work

This chapter does not mark the end of our venture; rather we can say that it is the beginning of a major endeavor that has been initiated. Naturally there are lots of activities left behind. In near future, the following experimental program will be executed

for detailed study and analysis of effectiveness of indigenous activated charcoal bed and heat exchangers. That experimental program includes:

- Computation of breakthrough curve by operating purifier for long duration with various concentrations of air and moisture impurities up to the level of 40%. The measurement of temperature, pressure, online purity readings, flow rate with data acquisition system must be carried out.
- Addition of flowmeter in low pressure line of compressor to measure the actual volume of suction gas.
- Study of impact of lubrication oil migration from high pressure compressor on purification.
- Impact of imperfect regeneration of adsorber bed and heat exchangers on purification.
- Study of adsorption property and other measurable properties with varieties of commercial activated charcoal available in India
- Actual helium flow rate and measurement of pressure drop in various components
- Measurement of LN_2 consumption
  - Quantity of LN_2 consumed per cycle of regeneration
  - Quantity of LN_2 consumed per nm^3 of output pure helium as a function of input impurity
- Study of time and power input for regeneration
- World's leading manufacturers are making PLC based fully automatic cryogenic purifier with the option of remote monitoring and user friendly graphical representation. Hence, it can be presumed that the valuable experience gained with the design and fabrication of this purifier and associated subsystems will result in the generation of a knowledge base that will enable us to design a entirely automatic unit and to achieve commercial success in India in the days to come.

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APPENDIX I

Specification of Purchased Components

Brief technical specification of all the valves and filters used in helium purifier are as follows:

Sl. No.	Valve Type & Designation	Brief Description
1	Ball Valve BV 1, 2, 3, 4, 5, 7, 10	On-Off (2 way), straight pattern, 6mm OD swagelok tube fittings end connection, pressure rating:206 bar, One piece SS 316 body, top loaded design, panel nut,
2	Ball Valve BV 9	Straight pattern, pressure rating 1000 psi, investment casting, 15% glass fibre filled reinforced PTFE seat, PTFE seal and packing, End connection: 3/8" NPT(Female)
3	Ball Valve BV 8	Straight pattern, pressure rating 1000 psi, investment casting, 15% glass fibre filled reinforced PTFE seat, PTFE seal and packing, End connection: 1" NPT(Female)
4	Check Valve NRV5	Fixed cracking pressure, SS 316 poppet, Nominal cracking pressure 1/3 psi, Downstream pressure at 20^{o}C :13.7 bar, 1" NPT(F) end connection,
5	Check Valve NRV 1, 2, 3	Fixed cracking pressure, SS 316 poppet, Nominal cracking pressure 1/3 psi, Downstream pressure at 20^{o}C :413 bar, 6mm compression fitting end connection,
6	Safety Relief Valve SRV 1,2	High pressure proportional relief valve, ¼" NPT(M) inlet and ¼" NPT(F) outlet end connection, Set cracking pressure 125 bar.
7	Safety Relief Valve for LN_2 vessel SRV3	Bronze body, metal to metal seated, closed bonnet, Set pressure range 0.20 to 40 bar, Pressure set at 0.50 bar(g), End connection: male thread type G 1"x1 1/4" BSPP, Discharge capacity of air at 0^{o}C and 1013.25 bar is 88 m^3/hr, Temperature range: -196^{o}C to 185^{o}C
8	Burst Disk BD	Tension loaded pre-bulged solid metal rupture disk. Burst pressure is 15 psig at 22^{o}C, Size is 2"
9	Needle valve NV 1,2,3	SS316 Integral Bonnet type, Working pressure at 37^{o}C is 344 bar, C_v:0.73, End connection: 6mm OD compression fitting
10	Micron Filter F 1, 3	Inline micron filter having replaceable sintered element of 7 micron nominal pore size, SS316 sintered element, 6 mm OD swagelok compression fitting end connection
11	Solenoid valve SV 1	Cryogenic type, Working temperature: LN2, vValve body: Brass, Operation: Normally closed, Coil supply: 240 V 50 Hz AC, Max. pressure: 2 bar(g), Coils have faston terminals, End fittings: 10 mm BSP (female)
12	Back Pressure Regulator BPR	Provides consistent back pressure control, Piston type, Pressure control range: 0-200 bar(g), Body material: SS316, Port: 6 mm Female NPT inlet and outlet,

Technical Data of the reciprocating compressor unit

Model No.	Series 1000B - He
Make	Metec Corporation, Korea
Dimension (L x W x H, mm)	1100 x 600 x 800
Net weight, kg	235
Number of stages	3
Number of cylinders	3
Cylinder bore (1st stage), mm	100
Cylinder bore (2nd stage), mm	36
Cylinder bore (3rd stage), mm	14
Piston stroke, mm	40
Compressor speed, rpm	1180
Intermediate pressure 1st stage, bar(g)	10 to 12
Intermediate pressure 2nd stage, bar(g)	50 to 55
Intermediate pressure 3rd stage, bar(g)	150
Oil capacity, l	2
Ambient temperature, oC maximum	48
Permissible inclination of compressor	10^{o}
Belt size, inch	A – 59 x 2EA
Pulley size, inch	8
Applied standard,	ISO 9001 / KS B 6236
Motor power, HP	10
Motor Electric power	400 V, 50 Hz
Motor speed, rpm	1455
Motor pulley size,inch	6.5
Motor weight, kg	70
Motor front bearing number	6206zz
Motor rear bearing number	6208zz
Motor make	Siemens

APPENDIX II

Drawings

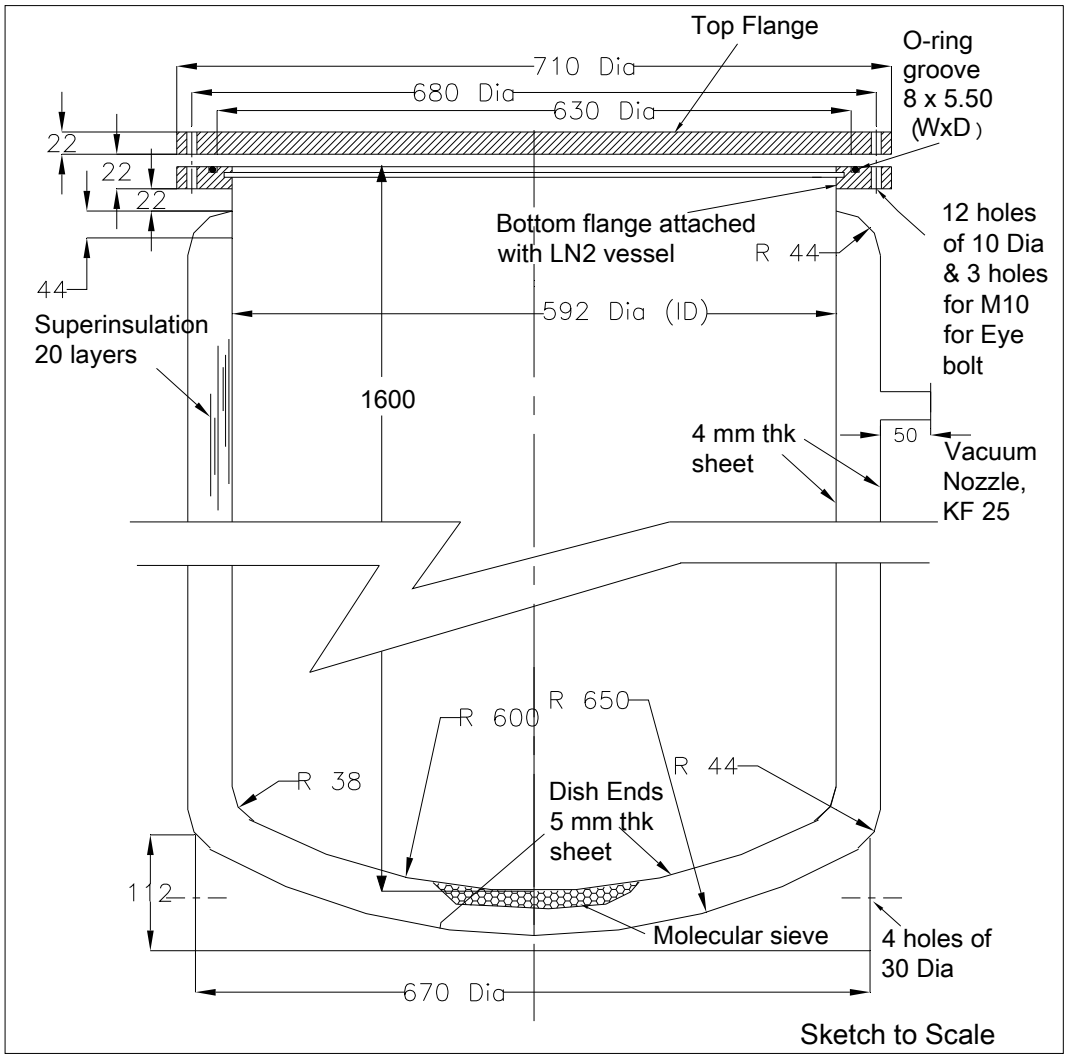

Fig. A1: Superinsulated LN_2 vessel of helium purifier

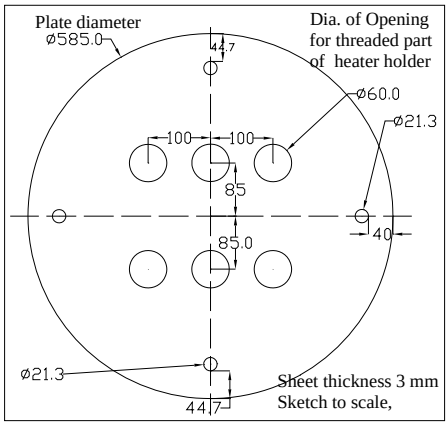

Fig. A2: Top plate of the cage of helium purifier

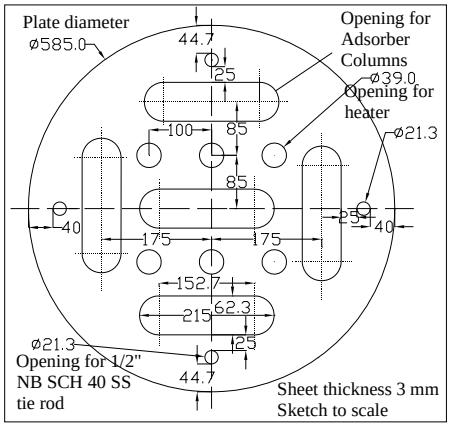

Fig. A3: Middle plate of the cage of helium purifier

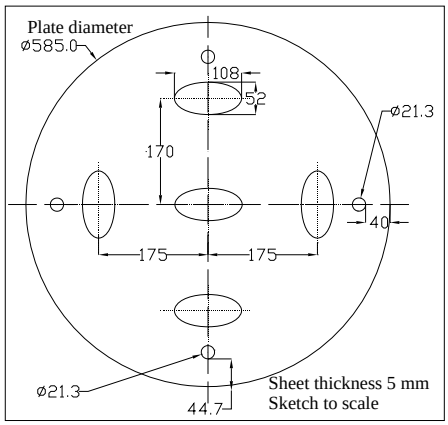

Fig. A4: Bottom plate of the cage of helium purifier

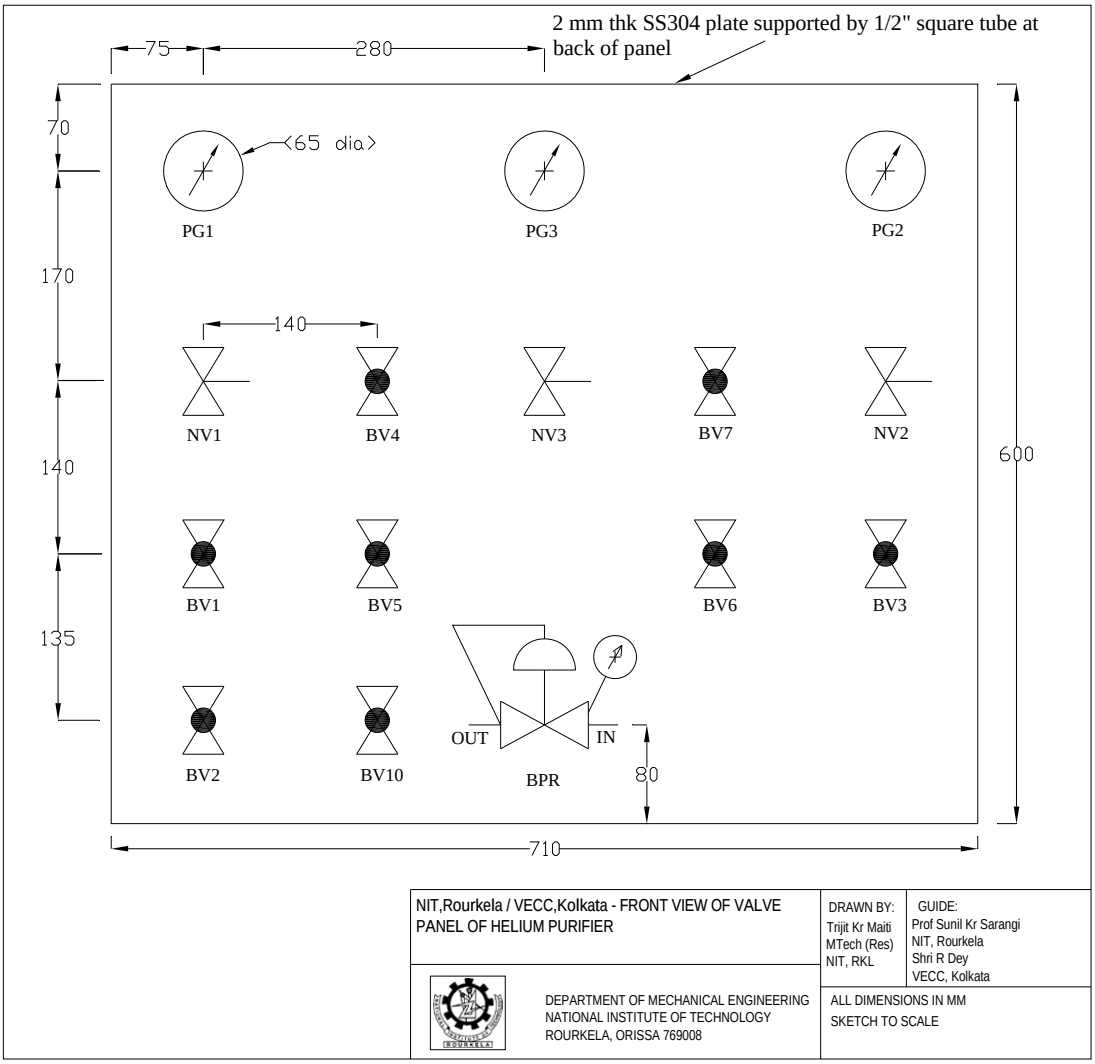

Fig. A5: Valve panel of helium purifier

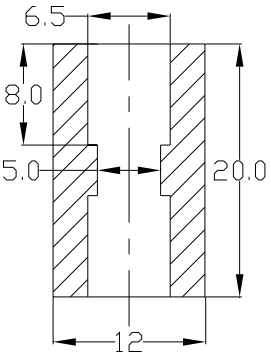

Fig: Short piece 6 x 6mm

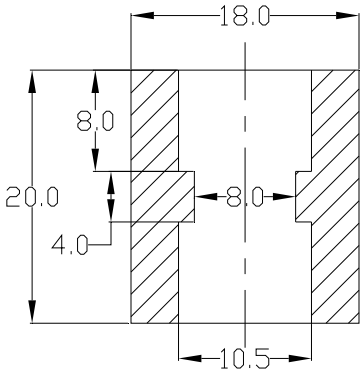

Fig: Short piece 10 x 10mm

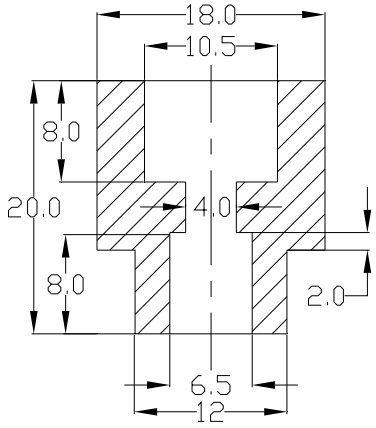

Fig: Reducer 6 x 10mm

Material: Stainless Steel 316L

Fig. A6: Tube connectors for socket welding for components within LN_2 vessel of helium purifier

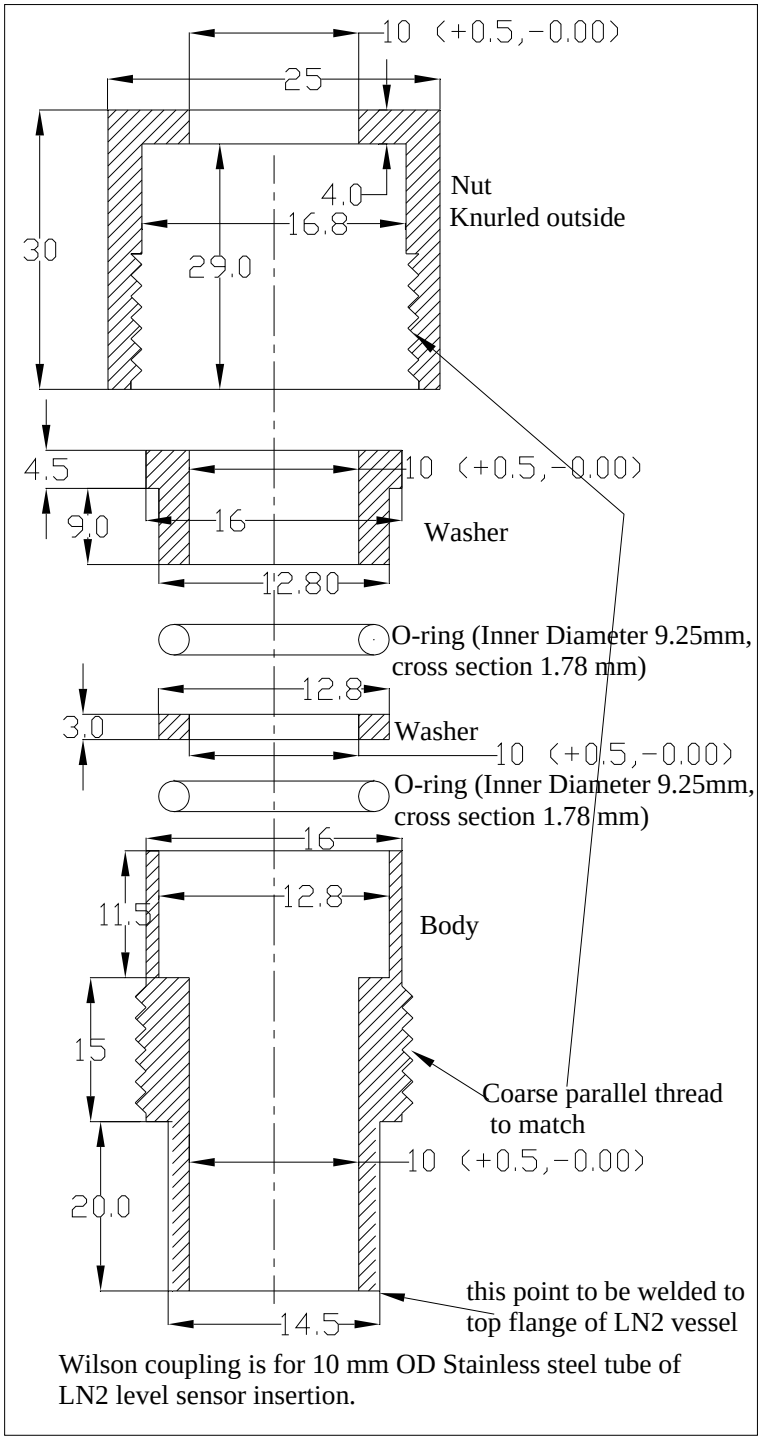

Fig. A7: Exploded view of Wilson Coupling used for LN_2 level sensor of helium purifier

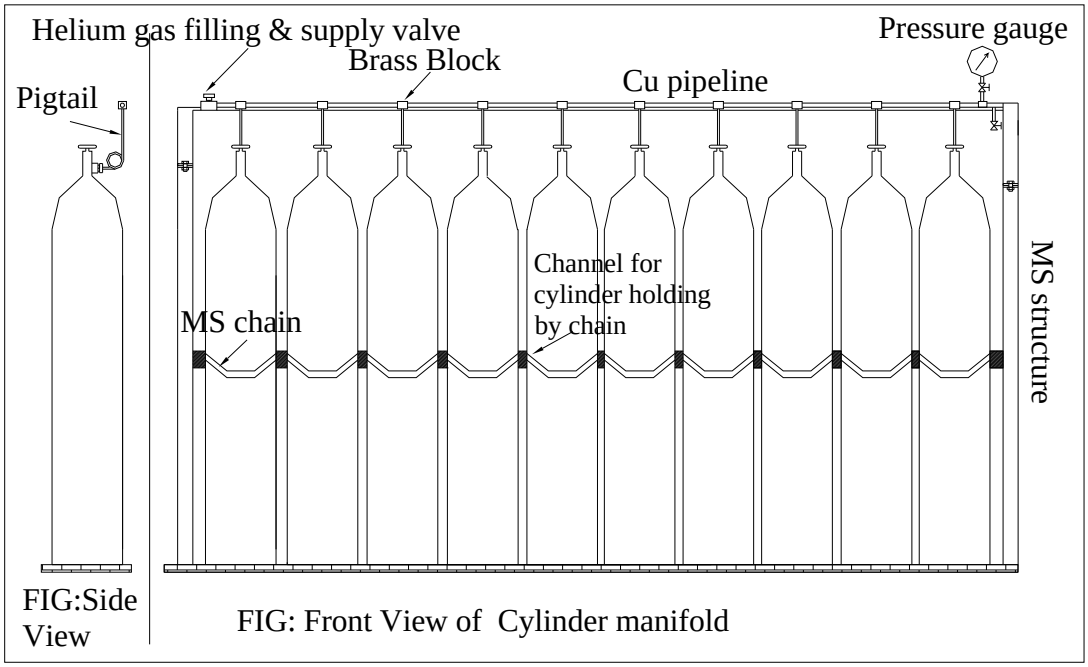

Fig. A8: Pure and impure cylinder manifold

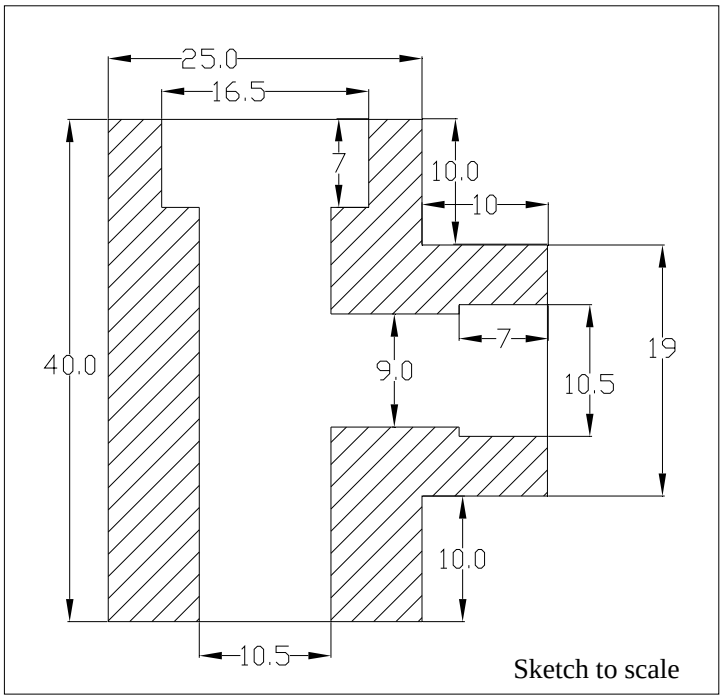

Fig. A9: End connector of tube-in-tube heat exchanger

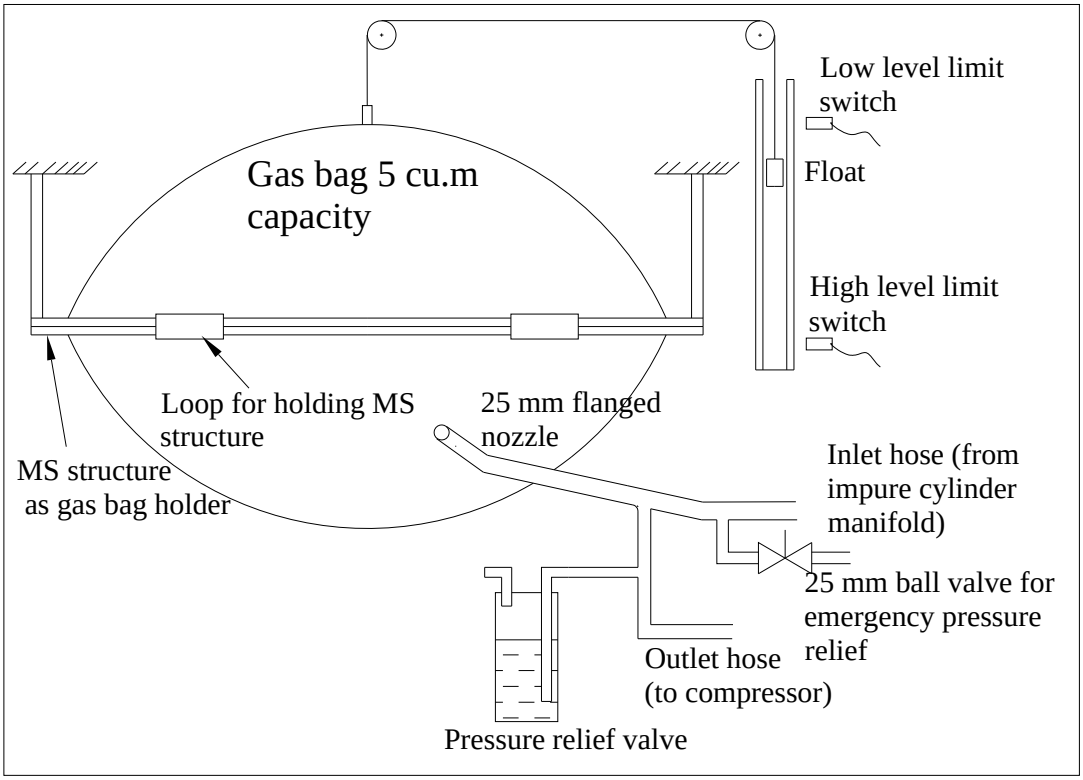

Fig. A10: Layout of gas bag system

Curriculum Vitae

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2000 – Till Date	**Scientific Officer**, Variable Energy Cyclotron Centre, Kolkata

Publications:

1. **Maiti, T.K., Dey, R., Sahoo, R.K., Sarangi, S.K** Development and Study of an Indigenous Helium Purifier based on Low Temperature High Pressure Adsorption of Impurities, *23rd National Symposium on Cryogenics*, National Institute of Technology, Rourkela, (2010)
2. **Pal, S., Panda, U., Mukherjee, A., Maiti, T.K., Dey, R** Experiences in the commissioning of new helium liquefier at VECC and the respective remedial actions, *23rd National Symposium on Cryogenics*, National Institute of Technology, Rourkela, (2010)
3. **Maiti, T.K., Dey, R., Banerjee, S., Mukherjee, A., Panda, U.S., Datta, N., Parate, J., Das, A** Development of cryoadsorber based helium purifier at VECC, Kolkata**,** *Indian Journal of Cryogenics*, Special Issue - Vol.2, (2005)
4. **Maiti, T.K., Dey, R., Banerjee, S., Mukherjee, A., Panda, U.S., Datta, N** Gas Management System for Liquid Helium Plant, *Proceedings of the National Seminar & Conference on Cryogenics and its Frontier Applications,* B.E. College, Howrah, West Bengal, (2004)