PERFORMANCE OF ADSORBER BED FOR DIFFERENT PARAMETER WITH HELIUM AT VERY LOW TEMPERATURE

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Abstract: The HRL plant at Very temperatures Among these, the purifier which removes H2 and Ne from helium gas at very low temperature is an absorber bed. The prototype of this for about 1/2 of full scale (30 g/s helium flow with inlet impurity 100 PPM by volume) has been fabricated by an Indian manufacturer. To measure the performance of this purifier, a cryogenic test facility with closed loop helium circulation at higher operating pressure (between 1 and 14 bar) is planned. This will need the design of a closed loop cryogenic piping, valves, temperature fixtures, layout of room temperature and cryogenic components with proper supports. Required insulation has to be designed to minimize the external heat leaks to the cryogenic components, which otherwise can lead to significant error in performance measurement. The aim of the research paper to study and collect the required information for m dissertation work.

Keywords: Helium Purification System, Adsorber bed, low Temperature

Introduction: Prototype of adsorber bed is a model of an actual adsorber bed which will be used to test the adsorption capacity of different adsorbents & capability of the adsorber bed of removing impurity from the supplied Helium gas. Direct use of the adsorber bed in the Indigenous Helium Plant might cause the problem of the inaccuracy and ineffectiveness in purifying helium gas & if it may not be capable to purify helium gas at required level of purification of 1PPM output than that might be more expensive to correct that design and implement new corrected bed during the operation. So, before applying that adsorber bed in the Indigenous Helium plant, it is essential to use the prototype adsorber bed. This prototype will work on the same principle and Low temperature of the actual one. To test the adsorber bed, the test facility should necessitate to develop. The proposed design of adsorber bed test facility is shown in fig.1.4. This adsorber bed test facility contain one adsorber bed, one filter element and two heat exchangers:

(1) Plate and Fin type Heat exchanger

(2) L He bath type heat exchanger.

Literature Survey:

[2.1] Helium contamination through polymeric walls by M.Gabal, M.P. Lozano, A.Ocac, M.P. Pinaa, J.Seséa,C. Rillo in ICEC 25–ICMC 2014

A typical configuration for a helium recovery plant is shown in Fig.2.1. An undetermined number of cryostats and transport Dewar are connected to the metallic pipes of the recovery line that could be extended over several labs located on different buildings. The evaporated helium flows through the recovery line into the gas bag that acts as a temporary storage buffer. In medium-pressure recovery systems without gas bag and with only metallic pipework, the total content of impurities can be lowered to tens of ppm. In those cases, the helium gas can be cleansed by liquid nitrogen traps or heated getter systems [2]. In high-pressure recovery systems, with a compressor and gas bag, a more powerful purification step is needed.

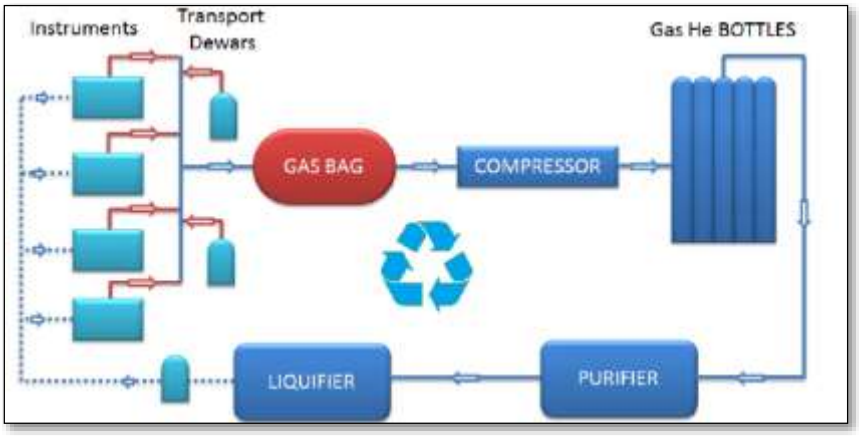

Fig.2.1.1 Scheme of a general liquefaction-recovery helium plant. Blue lines: metallic pipes, blue dashed line: Liquid helium transfer, red line: helium gas connection pipes to recovery line, material in study at this report.

Consequently, a purification step is mandatory due to the fact that He purity is the determining performance factor for any liquefier. In medium-pressure recovery systems without gas bag and with only metallic pipework, the total content of impurities can be lowered to tens of ppm. In those cases, the helium gas can be cleansed by liquid nitrogen traps or heated getter systems [2]. In high-pressure recovery systems, with a compressor and gas bag, a more powerful purification step is needed. The result shows that the helium in the storage bottles is typically 99.92 % pure, and the main impurities are in fact nitrogen and oxygen from the atmosphere. The impurities composition is 63.01 % nitrogen, 36.95 % oxygen and 0.05 % carbon dioxide. As it was expected, the N2:O2 molar ratio notably differs from the air value, 1.7 versus 3.7. This observation agrees with the differences in permeation behavior of nitrogen and oxygen through polymers [7].

2.1.2 Polymeric pipelines characterization:

For the experiments, a bundle of bottles containing high purity helium (99.999%) have been connected to a forward pressure controller to ensure 1448 mbar as absolute pressure upstream. Different pipelines, varying length, diameter and wall thickness and polymer nature have been tested at helium flow rate of 0.4 slpm a typical value for the evaporation rate in a transport Dewar.

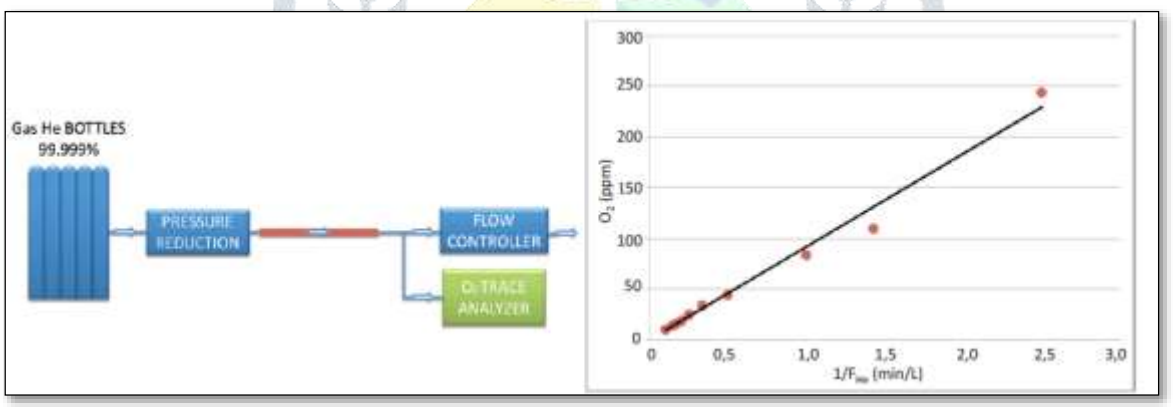

Fig.2.1.3. Evaluation of O2 contamination due to polymeric pipelines as a function of the helium flow rate: left) experimental set-up; right): experimental results.

[2.2] Purification of recovered helium with low level of impurities: evaluation of two different methods by M.P. Lozanoa*, M. Gabalb, J.Seséb , M.P. Pinab, C. Rillo ICEC 25–ICMC 2014

It is possible to achieve a low level of impurities by using the proper materials and procedures on the road to helium recovery. A comparison of two different methods applied for the purification of recovered helium with low level of impurities is reported in this paper. One method is the use of liquid nitrogen traps and the other one is the application of a purification system based on getter materials. The two methods tested to eliminate low level impurities in recovered helium gas are implemented in the Medium Pressure Helium Recovery Plant (MP-HRP) [5]

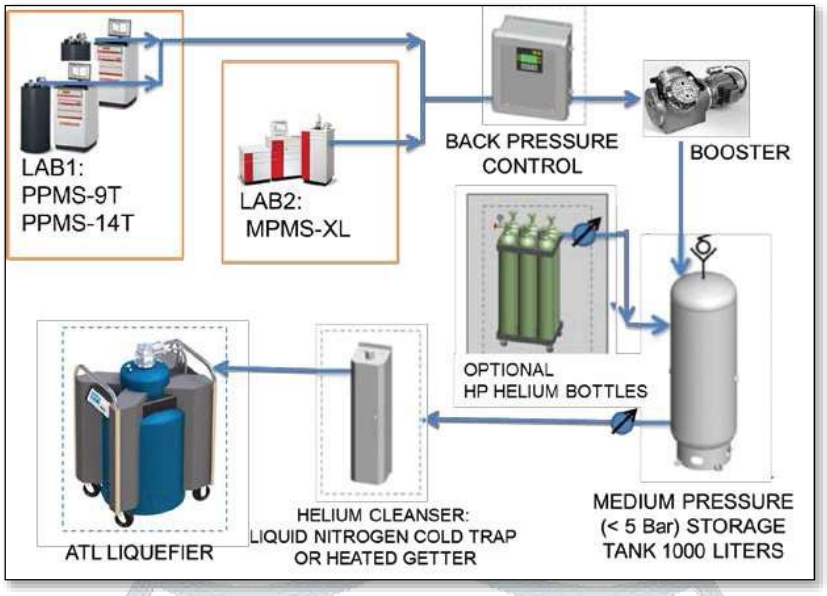

Fig.2.2.1. MP-HRP implemented to recover helium boil-off from PPMS and MPMS instruments. The circle indicates the position of the two cleansing methods tested in this work.

This is critical to keep oxygen content below 10 ppm. A back-pressure control system maintains the pressure seen by the instruments at a programmable constant value, typically 1 bar absolute with variations below 0.1%. The ATLs are fed by helium gas coming from the storage tanks, after passing through a helium cleanser, liquid nitrogen trap or heated getter. The cleanser purifies the helium to ultra-high purity grade (impurities < 0.5 ppm of O2) in order to guarantee the maximum liquefaction rate of the ATL liquefiers. High purity reaches the liquefier to take profit of its maximum liquefaction capacity when needed.The commercial helium gas of certified quality N50, i.e. 99.999 % of purity with < 2 ppm O2 guaranteed, provided in a bottle of 50 liter volume at 200 bar, was also measured. The average values of the obtained results are shown in table 2.1.

Helium gas analyzed	O2 (ppm)
He N50	0.75
LN2 cold trap	0.57
Heated Getter	0.02

Table 2.2.1. Oxygen contents in helium gas.

[2.3] Improvement of the operational settings of a helium purifier, leading to a higher purity of the recovered gas by Hiroshi Ikedaa, Yutaka Kondoa in ICEC 25–ICMC 2014

Normally, the internal purifier operating mode of a helium liquefier starts purification by going through the processes of purge, cool down 1, standby, and cool down 2 and then performs regeneration and goes into purge mode once again. As shown in the internal purifier flow diagram [4] in Fig. 1, the internal purifier operating condition of a helium liquefier is determined by adjusting the cold end temperature TI3475, cold flow F3410, regeneration completion temperature TI3465, and heater control output R3470. As a result, the recovered gas purity during regeneration improved from 33.7 to 65.0%. Thereafter, the recovered gas purity during regeneration did not improve even when the cold flow F3410 was increased. Next, we monitored the recovered gas purity during regeneration by fixing the cold flow F3410 to 180 ℓ /min and changing the cold end temperature TI3475. From this result, we were able to improve the recovered gas purity during regeneration from 33.5 to 99% by changing the cold end temperature TI3475 of the internal purifier from 32.5 to 22 K. Ultimately, the settings were set to cold end temperature TI3475=22.0 K, cold flow F3410=180 ℓ/min, regeneration complete temperature TI3465=145 K, and heater control output R3470=24%.

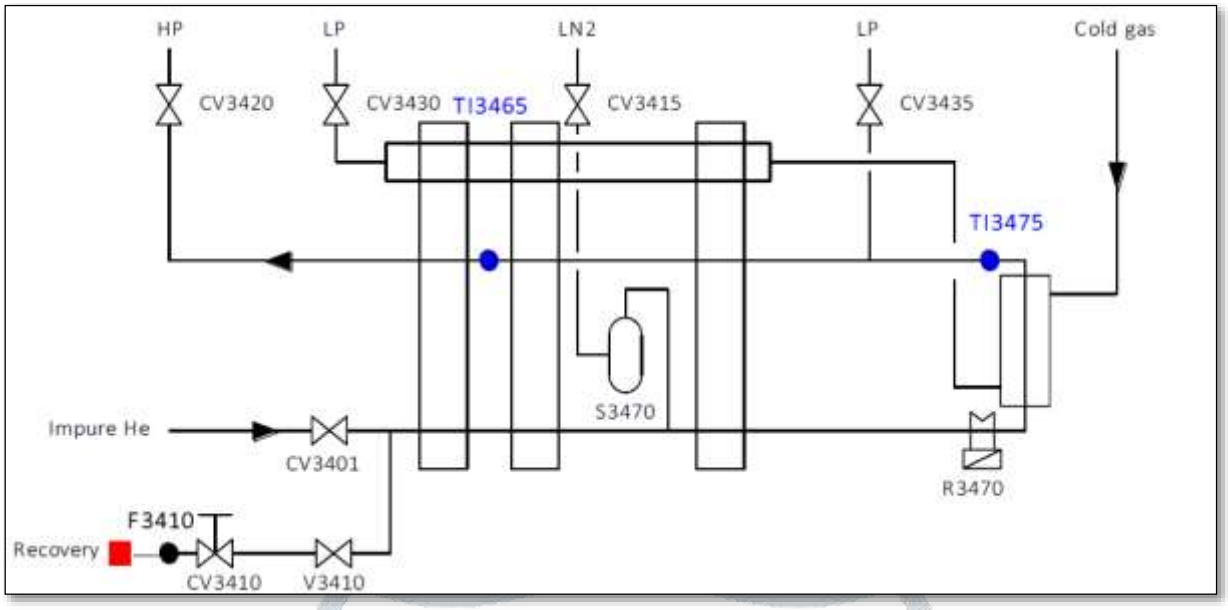

Fig. 2.3.1. Internal purifier flow for Linde L280 system.

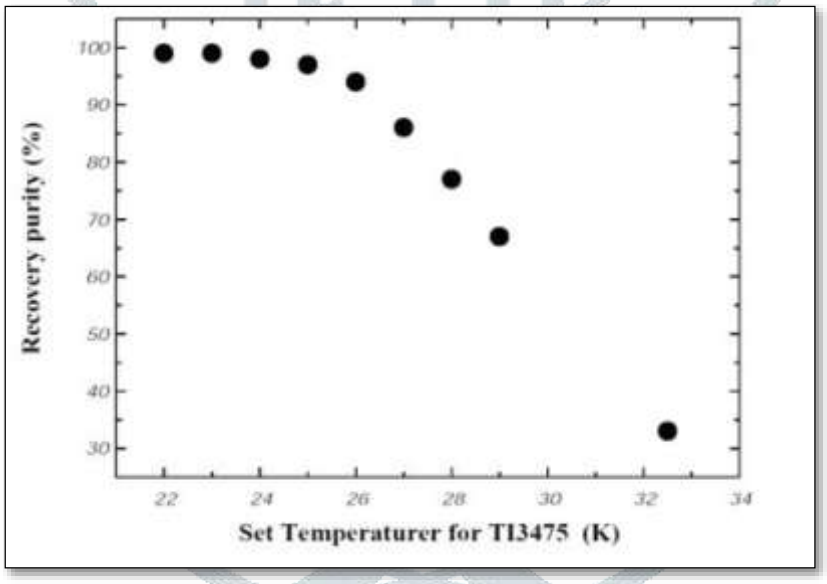

Fig. 2.3.2. Monitored the recovered gas purity during regeneration by fixing the cold flow F3410 to 180 ℓ /min and changing the cold end temperature TI3475.

[2.4] Porosity, Surface Area, Surface Energy, and Hydrogen Adsorption in Nanostructured Carbons by Alejandro Anso´n,† Jacek Jagiello, Jose´ B. Parra, M. Luisa Sanjua´n,| Ana M. Benito, Wolfgang K. Maser and M. Teresa Martez, J. Phys. Chem. B 2004, 108, 15820-15826

The nanostructured carbons such as nanofibers (NFs), multiwall nanotubes (MWNTs), and single-wall nanotubes (SWNTs) have been recently probed for hydrogen storage. Nitrogen isotherms at 77 K and CO2 isotherms at 273 K have been measured, and SWNTs have been characterized by Raman spectroscopy. The raw sample was synthesized in an electric arc reactor with graphite electrodes, using Ni/Y (2/0.5 atomic %) as catalysts.11 The as-obtained soot, which is collected from the inner walls of the reactor, contains bundles of SWNTs together with some impurities: metallic nanoparticles, amorphous carbon, and graphitic carbon.12 The raw material was oxidized with air in a furnace at 350 °C for 1 h, under static conditions. This material is here named the air-treated sample.

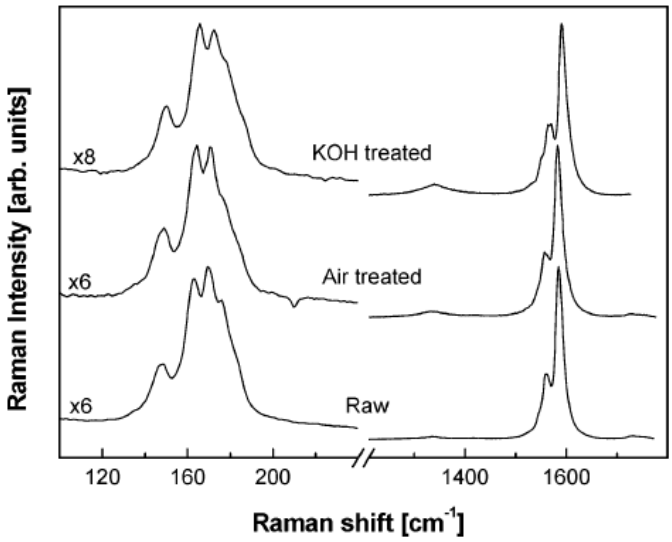

Figure 2.4.1. Raman spectra for the raw, air-treated, and KOH-treated samples.

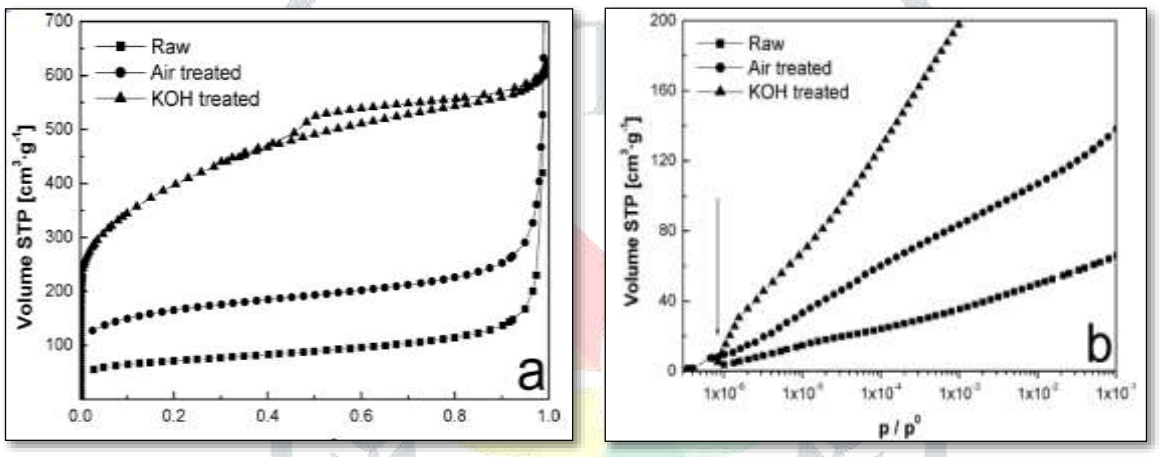

Figure 2.4.2. Nitrogen isotherms at 77 K for raw, air-treated, and KOH treated samples

Figure 2.4.2a shows the nitrogen adsorption isotherms at 77 K for the three samples. The raw sample exhibits a type II isotherm, according to the IUPAC classification. The isotherm presents an initial steep increase in adsorption because there are micropores in the raw sample. This fact has been previously reported for the same material.14 A narrow hysteresis loop (not shown in Figure 2) appears at very high relative pressures, around 0.9, together with the high increase in the adsorption. Figure 2.4.2.b is a semilogarithmic plot of the nitrogen isotherm that allows us to appreciate differences between the three samples at very low relative pressures. The KOH-treated sample adsorbs more nitrogen at almost any pressure, but around 10-6 the isotherm crosses the isotherm of the air-treated sample. Although in the limiting zone of the very low relative pressures the precision of the measurement devices becomes worse.

[2.5] Design of the Helium Purifier for IHEP-ADS Helium Purification System by Zhang Jianqin, Li Shaopeng, Zhang Zhuo, Ge Rui , Institute of High Energy Physics, Chinese Academy of Science, Beijing, 10049, China

The impure helium is mainly from the tests of superconducting cavities and the failures of the cryogenic system. The impurities of the impure helium can solidify at low temperature, which will choke the tubes and damage the turbo expanders. The helium purification system can keep the purity of helium for 99.9995% and has a storage capacity of 20000Nm3 (including impure helium and pure helium) at 20MPa. The simplified process scheme of the purification system is in Fig.1, which includes gas bag, compressor, coalescing filters, dryer, helium purifier and high-pressure cylinder manifold.

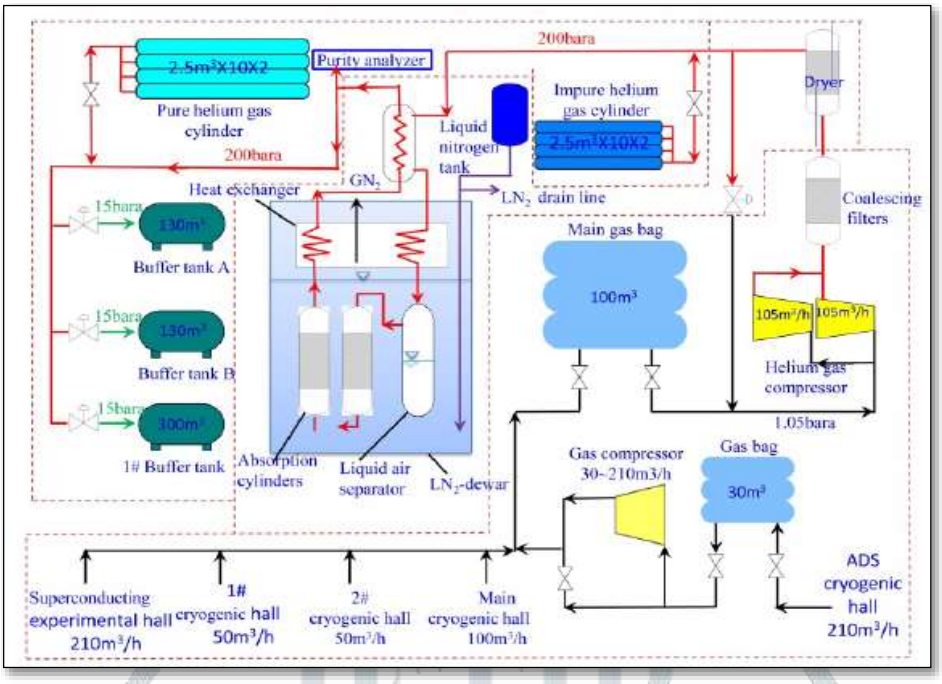

Fig.2.5.1 Simplified scheme of the purification system

Helium purifier is a key equipment of the helium purification system. Some countries have built their own high-pressure purifiers [1, 2]. The purifier works under the condition of 77K, 20MPa, which mainly includes high pressure heat exchanger, liquid air separator and carbon activated adsorption cylinders. In this work, the working principle and the design of the helium purifier are presented, and the studies of heat exchanger and the adsorbents are carried out.

2.5.2. Design of helium purifier:

The height of the purifier is 3050mm, and the diameter of Dewar is 600mm with a thickness of 5mm. The outer heat exchanger acts as dryer which brings down the water dew point to approximately 1℃. The helically coiled tube-in-tube heat exchanger is selected as the high-pressure heat exchanger, which is compact and high efficient. The outside diameter of the inner tube is 8mm with a wall thickness of 1.5mm, while the outside diameter of the outer tube is 16mm with a wall thickness of 3mm.The diameter of the coil is 270mm. In order to decrease the drop pressure of the tube, the heat exchanger is divided into five layers. The result of the high-pressure heat exchanger is shown in table 2 and the tube length is 11.84m. The liquid air separator (vol. 18.3l) collects the liquid air at the bottom of the separator.

	Input temperature (K)	Input Pressure (MPa)	Flow rate (g/s)	Output temperature (K)	Pressure drop (Pa)
Input gas	300	20	5	93.5	5140
Output gas	78	19.8	4.9	295	2360

Table 2.5.2 Parameters of the helically coiled tube-in-tube heat exchanger

[2.6] cryogenic adsorber design in a helium refrigeration system by Hu Zhongjun1, Zhang Ning2, Li Zhengyu1, and Li Q, American institute Of Publication in 2012

The refrigeration cycle protection is achieved by the cycle cryogenic adsorbers, usually operating at about 80 K (air) and 20 K (H2 and Ne). The purification of helium to be re-liquefied is achieved by the external or internal purifier. Cryogenic adsorption is a similar process to drying, but at the cryogenic temperatures. After the cryogenic adsorption, each impurity components (H2O, N2, O2, Ne, H2, CxHy) should be less than 1ppm [1]

2.6.1 cryogenic adsorption mechanism

As shown in Figure 2.6.1, the adsorption capability of nitrogen on activated charcoal at 77 K will increase when the gas pressure increases [2]. When pressure or temperature changes, the adsorbents can trap more or release the impurities. When the nitrogen partial pressure is greater than 1 mbar, the adsorption capability is greater than 100 Ncm3/g. In the dynamic adsorption process, the adsorbent's bed is divided into three sections, the saturated adsorbent zone, the adsorption mass transfer zone and the clean adsorbent zone.

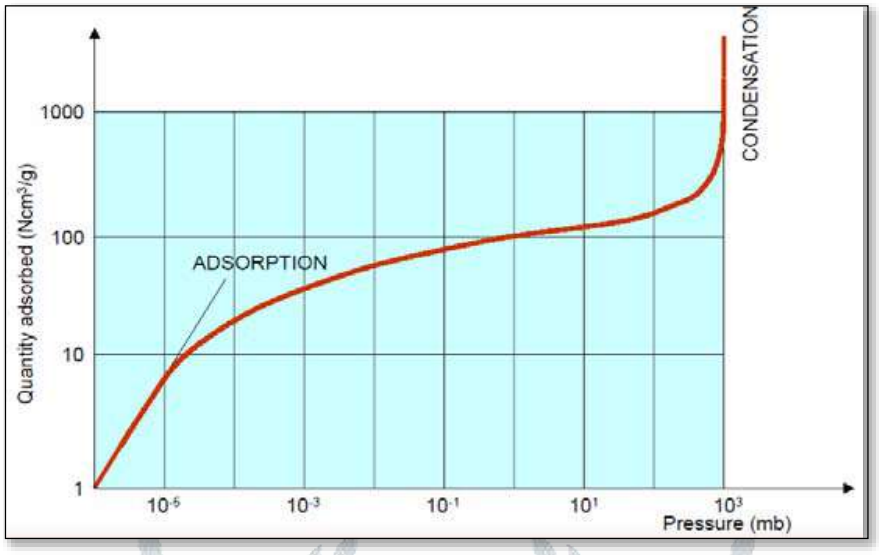

Fig 2.6.1 Adsorption of nitrogen on activated charcoal, at 77 K.

2.6.2 Cryogenic adsorber design:

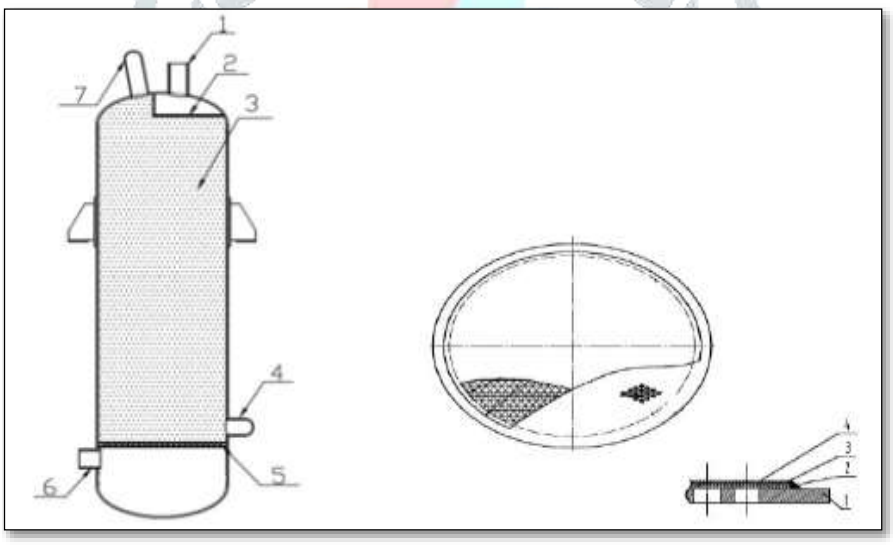

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Figure2.6.2 Structure scheme of a cryogenic adsorber. (In Figure 2a, 1 is the gas entrance, 6 is the gas exit, 3 is the adsorbent bed, 4 and 7 are filling holes, 2 and 5 are the filters.

Shown in figure 2.6.2.a, the structure of the designed cryogenic adsorber is described. The gas stream usually flows into the cryogenic adsorber from the top and then flows out bottom. But the gas flow direction is adverse when the active carbon is regenerated. figure 2.6.2b describes the detailed structure of the filters. The filter to block the activated carbon dust consists of four porous layers.

References:

1 **Helium contamination through polymeric walls"** by M.Gabal, M.P. Lozano, A.Ocac, M.P. Pinaa, J.Seséa,C. Rillo in ICEC 25–ICMC 2014

2 **Purification of recovered helium with low level of impurities: evaluation of two different methods"** by M.P. Lozanoa*, M. Gabalb, J.Seséb , M.P. Pinab, C. Rillo ICEC 25–ICMC 2014

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4 **Porosity, Surface Area, Surface Energy, and Hydrogen Adsorption in Nanostructured Carbons"** by Alejandro Anso´n,† Jacek Jagiello, Jose´ B. Parra, M. Luisa Sanjua´n,| Ana M. Benito, Wolfgang K. Maser and M. Teresa Martı´nez, J. Phys. Chem. B 2004, 108, 15820-15826

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