HIGH CAPACITY HELIUM PURIFIER FOR 14 G/S AT 200 BAR

H. Neumann¹, G. Perinic²

Abstract
The purifier comprises two independent purification lines. Each line is subdivided into three purification stages which remove the impurities oil with coalescing filters and an active charcoal filled adsorber, moisture with a molecular sieve bed and oxygen and nitrogen by condensation in a phase separator and consequent adsorption in a silicagel bed both at liquid nitrogen temperature. The purifier is designed for a throughput of 14 g/s at 200 bar in continuous operation. In non continuous operation, the purifier can be run with a throughput of up to 28 g/s. The purified helium has an impurity content of less than 1 ppm.

¹ Institut für Technische Physik, Forschungszentrum Karlsruhe, Postfach 3640, D-76021 Karlsruhe, Germany
² LHC Division

Presented at the Eighteenth International Cryogenic Engineering Conference (ICEC 18)
21-25 February 2000, Bombay Mumbai, India
The purifier comprises two independent purification lines. Each line is subdivided into three purification stages which remove the impurities oil with coalescing filters and an active charcoal filled adsorber, moisture with a molecular sieve bed and oxygen and nitrogen by condensation in a phase separator and consequent adsorption in a silicagel bed both at liquid nitrogen temperature. The purifier is designed for a throughput of 14 g/s at 200 bar in continuous operation. In non continuous operation, the purifier can be run with a throughput of up to 28 g/s. The purified helium has an impurity content of less than 1 ppm.

INTRODUCTION

The 300W-1.8K-refrigerator described in [1] and the 2kW-4.4K-refrigerator described in [2,3,4] of the Institute for Technical Physics (ITP) supply experiments of the ITP and several other Institutes of the Research Centre Karlsruhe with more than 7000 h of refrigeration and more than 200,000 l of liquid helium per year. The cryogen helium is distributed and recovered via a widely branched transfer line system. For the liquefaction process the helium must fulfil very high purity standards. Due to the pollution of the helium introduced by the experiments (impurity: air), by the storage (impurity: air and moisture) and by the recovery compressors (impurity: oil) a high capacity and high quality purification system is essential.

In 1996 the old purification system built in 1969 has been replaced by a state-of-the-art fully automatically operated purification system. The paper presents the purifier layout and reports the experience gained in the specification, manufacturing and commissioning process as well as the experience in three years operation.

PLANT DESCRIPTION

The new helium purifier consists of two identical and completely independent purification lines. Figure 1 shows one line consisting of three purification stages.

In the first stage oil impurities are removed in two steps. First two coalescing filters (vol. 0.5 l) in series extract oil droplets. Then the helium stream is led through an active charcoal filled adsorber (vol. 56 l) which removes remaining oil droplets and oil vapour.

In the second stage moisture is extracted by the means of a molecular sieve bed (vol. 56 l) and finally in the third stage oxygen and nitrogen are removed. The third stage comprises two parts both at liquid nitrogen temperature, first a condensation stage which reduces the nitrogen and oxygen content down to their respective vapour pressure and secondly a silicagel adsorption bed (six cylinders – each with 56 l). Identical vessels have been chosen for the oil adsorber, the drier, the low temperature separator as well as the low temperature adsorbers in order to reduce manufacturing costs.
The purifier is designed for continuous operation with a throughput of 14 g/s at 200 bar which is ensured by an alternating operation of the two purification lines. On the operational line, the low temperature condensation stage will fill up with liquid nitrogen and liquid oxygen approximately every half hour in the worst case. It is therefore emptied automatically and online if a certain liquid level is reached. Furthermore, the first oil filter is automatically blown off every two hours to an oil sump. Both the drier stage and the low temperature adsorption stage are designed for an uptime of 12 hours which derives from the time required to regenerate the loaded purifier line. The other extraction stages have longer uptimes and are therefore maintained manually i.e. the second oil filter is checked at regular intervals and the active charcoal of the oil adsorber is foreseen to be replaced after five years of operation.

If necessary, either purifier line can also be run with a throughput of up to 28 g/s by which, however, the uptime is reduced and hence the operation of the purifier becomes discontinuous.

The regeneration of the operational purifier line is started by any one of the following five criterions:

- dewpoint temperature at the drier outlet in excess of -67°C
- nitrogen impurity at the centre of the last silicagel adsorption cylinder in excess of 10 ppm
- nitrogen impurity at the outlet of the purification line in excess of 10 ppm
- maximum uptime in excess of an operator defined time limit
- manual triggering of the regeneration by the operator

For the regeneration first the condensation stage is emptied and then the line is depressurised. The oil removal stage and the drier are depressurised together to the helium recovery system (gasometer). The gas from the low temperature stage is analysed during depressurisation and directed to the helium recovery system as long as the oxygen content stays below 5%. Above 5% oxygen content this gas is fed into the chimney. The oxygen content begins to rise at lower pressures due to the fact that the regeneration of the low temperature adsorbers is started by the pressure decrease.

After the depressurisation, the regeneration of the drier is achieved by heating up the adsorber bed to 100°C while flushing it with dry nitrogen. In the low temperature stage first the dewar in which the adsorber cylinders are submerged is emptied recovering the liquid nitrogen to the buffer tank. Then the regeneration is carried out by warming up the adsorber cylinders externally by the means of a closed circuit heating loop until 200 K are reached on the surface and in the centre of the adsorber cylinders. The reneg-
eration is completed by an evacuation of the low temperature adsorbers together with the drier and a pressurisation with pure helium.

The purifier is equipped with an automatically process control system (S5 115 U PLC from Siemens) and a local operator panel. The purification, the change-over between the two lines as well as the regeneration are controlled fully automatically, however, the operator can induce the various processes manually on the local panel or from a remote supervisory system [5].

COMMISSIONING

The purifier manufactured by L’Air Liquide was delivered in mid 1996 almost completely assembled on one skid. On site only the liquid nitrogen buffer tank, the evacuation pumps, the interface connections and the control cubicle had to be mounted and the control and power cables to be installed and connected.

The commissioning showed very soon that the purification process worked very well and fulfilled the specified requirements. However, it also became clear that the software required some improvement. In particular the loss of an important quantity of helium on one occasion which was due to an undiscovered faulty regeneration process configuration showed that a thorough analysis of the process logic in respect of plant safety should have been carried out prior to the programming.

Besides the software problems we also encountered a few hardware problems: The regeneration blowers had to be soundproofed in order to fulfil the severe regulations in the experimental hall. All safety valves had to be reset as the locking nuts of the adjustment screws had not been tightened correctly by the manufacturer and the set pressure decreased due to the vibrations of the plant. Furthermore, it was only perceived during operation that the length of the capillary from the inside of the last low temperature adsorber to the nitrogen trace analyser combined with the high operation pressure and the small flow through the analyser of 35l/h caused a time delay of the saturation signal of the adsorber of two hours. This problem was tackled by the installation of an 800l/h bypass of the analysers. In the process of solving the time delay problem, an improved logic for the nitrogen trace analysers was found and implemented. It uses the analyser of the regenerating line as additional backup analyser for the operating line taking samples at the outlet of the purifier.

OPERATION EXPERIENCE

After commissioning at the end of 1996 and the elimination of some technical faults during 1997 the purifier works quite well in continuous operation. The regeneration was usually started by the saturation of the low temperature adsorber. The other reasons for triggering the regeneration in the order of frequency were a dew point temperature of the drier exceeding the limit and the manual triggering of the regeneration by the operators. Experience shows that triggering of the regeneration by the dewpoint temperature alarm which means a reduced uptime of the drier indicates an incomplete previous regeneration of the drier. Triggering of the regeneration by the operator is mainly used just before a weekend to ensure the supervision by the operators or to enable revision works on one line.

Since the end of 1997 some new problems have emerged of which the most important ones shall be described here.

One particular problem are the leakages which have kept appearing on the numerous screwed joints. On the one hand leakages cause high helium losses at the pressure level of 200bar and on the other hand the resulting pressure loss in the stand-by purifier line has been found to cause a pressure wave going along with a very high flow rate upon putting the line into purifying mode. The high flow rate in the adsorber and the drier vessels do cause the whirl-up of the adsorbing material and hence the production of dust. This dust is transported into the piping and causes in consequence the blockage of the downstream filters being the inlet filters of the gas analysers, whose blockage leads to an error of the impurity measurement. The blockage of the filter F1 (see Fig. 1) prevents a correct regeneration of the drier as the adsorber is no longer warmed up evenly and the desorbing moisture is not thoroughly extracted by the flush dry nitrogen gas. Water has been found in the piping underneath the drier and also in frozen form block-
ing the heat exchanger of the low temperature stage. The blockage of the filter F1 has furthermore caused a repeated burn out of the regeneration heater of the drier as the heat is not carried off by the nitrogen. The problem of leakages has been tackled by replacing some of the screwed joints by welded joints. Furthermore, a little vessel has been installed underneath the drier which collects condensing water and last but not least the software has been modified to allow permanent pressurisation of the standby line from the operational line.

Another important problem is the pollution of the analysers (multiple component detector WE3M-2 by Linde). The gas to be analysed is stimulated to glow due to an alternating current discharge in the measurement cell. The light intensity is proportional to the impurity content and this intensity is assigned to a photo-current by sensors consisting of an interference filter and a photodiode. The sensors are located on the outside of a window of the measurement cell. Experience shows that impurities do create deposits on the measurement cell window. Finally these deposits cause an incorrect measurement result of the gas pollution. An improvement of the analysers could be achieved by the installation of a light source within the measurement cell which is switched on at regular intervals while the alternating current discharge is switched off. This light source could be used as a test signal for a windows check.

CONCLUSIONS

After installation and lengthy repair of initial technical faults the new helium purifier works reliable. The main advantages of this new purifier in comparison with the older one are the fully automatically operated purification system and consequently less demand of manpower. The main problems could be attributed to leakages, obstruction of filters and pollution of the analysers. Future works concern the replacement of more screwed connections by welded connections to improve the tightness. Furthermore, it is foreseen to improve the reliability of the nitrogen trace analysis by the implementation of a windows check. Finally the control system shall be completely integrated into the cryogenic network control system of the Forschungszentrum Karlsruhe described in [5].

REFERENCES

4. Perinic, G. Experience in Four Years Operation of the 2kW He I-Refrigerator at FZK/Karlsruhe Advances in Cryogenic Engineering (1998) 43 565-569