Residual Oil Aerosol Measurements on Refrigerators and Liquefiers

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Abstract. The purity of the process gas is essential for the reliability of refrigerators and liquefiers. Filtration and adsorption of impurities like water, nitrogen, and oil result in a major effort, cost, and maintenance in the helium process. Expensive impurity monitors for moisture, nitrogen, and hydrocarbon contents are required to identify filter failures and leakage immediately during the operation. While water and nitrogen contaminants can be detected reliably, the measurement of oil aerosols at the ppb-level is challenging. We present a novel diagnostic oil aerosol measurement system able to measure particles in the sub-µm range. This unit enabled us to evaluate and improve the oil separation system on a LINDE TCF 50 helium liquefier.

INTRODUCTION

Helium liquefaction and refrigeration is based on the compression, expansion, and heat transfer with helium as a process fluid. Oil-injection screw compressors are used to compress helium of near atmospheric pressure to a high pressure level of typically more than 10 bar. A multi-stage oil removal system (ORS), installed downstream of the compressor, separates the oil from the process gas (Fig. 1). To prevent the coldbox, in particular the installed heat exchangers and expansion turbines, from oil contamination it is necessary to provide purity in the range of ppb. The high mass flow of the process gas and the long term operation of the plant can lead to significant oil intrusion if the oil removal system is not able to provide the required separation efficiency. The result is a blockage of the heat exchangers followed by a rapid loss of cooling capacity and, in the worst case, damage of the expansion turbines.

Most LINDE standard helium plants are equipped with a LINDE Multi Component Detector (MCD) in combination with a pyrolizer. The instruments are installed between high and low pressure line to monitor N_2, H_2O, and oil (hydrocarbon) impurities constantly. As experience of Helmholtz-Zentrum Berlin with a LINDE TCF50 has shown, the N_2 and H_2O impurities are detected very reliably. But although the monitored oil level was zero, traces of oil were still found on specific points in the high pressure line. These points were the metal mesh cold box inlet filter and a ball valve downstream of the oil removal system (Fig. 2).

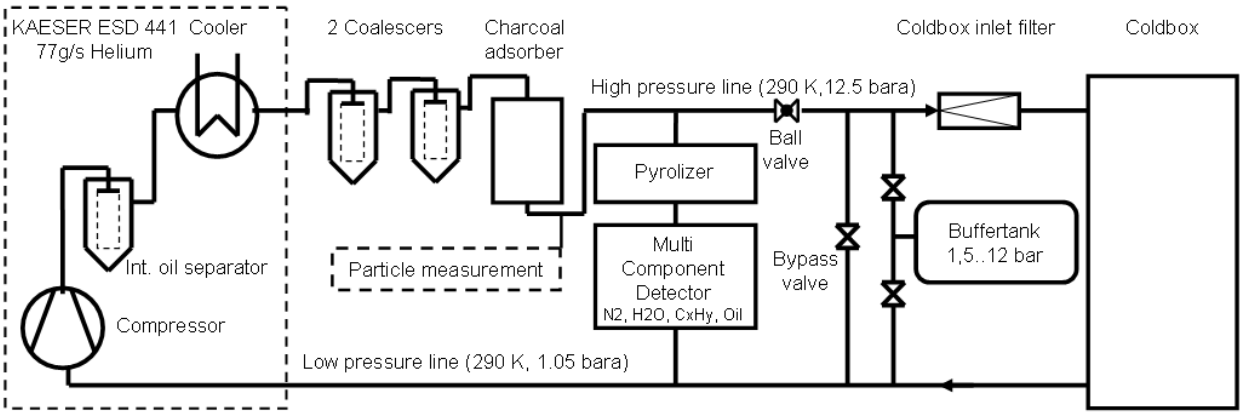
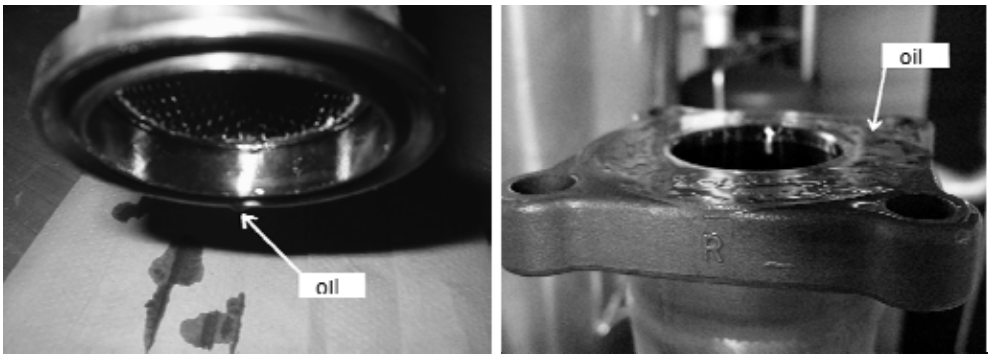


FIGURE 1. Simplified schematic of the LINDE TCF50 helium plant at HZB.



(a) (b)

FIGURE 2. Oil residues inside the high pressure line of a LINDE TCF50 liquefier. (a) Coldbox inlet filter, (b) Flange of the ball valve downstream the charcoal adsorber.

AEROSOL PARTICLE MEASUREMENT SYSTEM

Optical instruments for particle measurements are widely used in industrial air quality control, environmental monitoring, and clean room technology. The principle of counting and size measurement of airborne particles is shown in Fig. 3. The aerosol to be tested is passed through an illuminated zone and scatters the light of a laser beam in all directions. Part of this scattered light in a certain acceptance angle is collected by the optical detector. The amplitude of the photoelectric signal generated by the crossing particles is compared with standard calibration curves obtained from known particles [1]. This technique enables one to count the number as well as to classify the size of the particles in the range from sub micrometer to some micrometers.

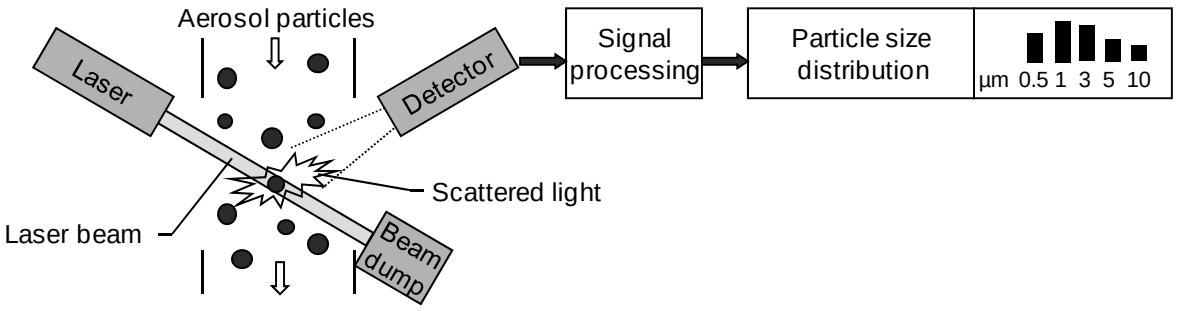


FIGURE 3. Principle scheme of an airborne particle counter.

A TSI AeroTrak 9110 portable airborne particle counter was used for oil aerosols measurements. The main parameters of the AeroTrak 9110 are displayed in Table 1.

TABLE 1. Parameters of the TSI AeroTrak 9110 [2].

Size range	0.100 µm to 10 µm
Particle Channel Sizes	0.1, 0.15, 0.2, 0.25, 0.3, 0.5, 1.0, 5.0 µm
Size resolution	<15 % @ 0.2 µm (per ISO 21501-4)
Counting Efficiency	50 % at 0.100 µm; 100% for particles > 0.15 µm (per ISO 21501-4 and JIS)
Concentration Limit	100,000 particles/ft³ (3,500,000/m³) @ 10 % coincidence loss
Flow Rate	1 CFM (28.3 l/min) +/- 5 % accuracy (Air)
Size/weight	23.6 cm x 20.6 cm x 52.2 cm / 12.7 kg with battery

Since the device was designed for clean room applications, the measuring cell can only withstand atmospheric pressure. Hence the particle counter cannot be directly connected to the high pressure helium line. A high pressure diffuser (HPD) was used to reduce the inlet pressure isokinetically (Fig. 4). The PMS HPD II-100 is based on cascaded nozzles to make sure that no particles are held back or are produced inside the pressure reducing device. The waste gas is exhausted through a HEPA filter to atmosphere. The inlet pressure of the HPD is specified by the manufacturer to be from 2.7 to 7.9 bar [3]. For the measurements on the TCF50 helium plant, the HPD was used with the typical high pressure of 12.5 bar. No damage or adverse effects on HPD or particle counter were detected.

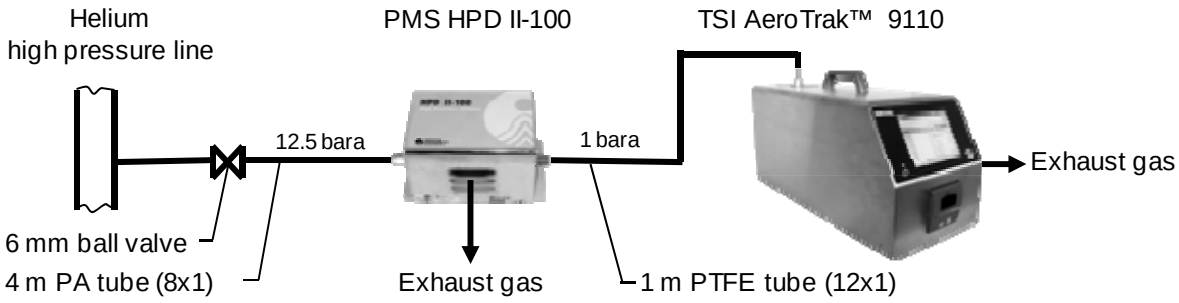


FIGURE 4. Particle measurement setup.

The flow rate and internal measurement of the TSI AeroTrak 9110 is specified and calibrated with air to 1 cubic foot per minute (CFM) or 28.3 l/min. A factory calibration for helium was not available. As a workaround, the measurement procedure for helium was predefined to a fixed measurement duration of 1 min. An additional measurement of the exhaust gas of the particle counter with a float type flow meter (Meister Stroemungstechnik flow monitor 5 - 50 Nl/min helium) resulted in a helium gas flow of 15 Nl/min.

MEASUREMENTS

The LINDE TCF50 oil removal system was measured and the results were compared with the values of two other liquefiers available at HZB. A LINDE L700 and a LINDE L140 were used as a reference. Each plant is equipped with a set of three coalescer filters installed as a precaution (Table 2).

The measuring point was located on each plant immediately downstream of the charcoal adsorbers, close to the inlet T-fitting of the regular Multi Component Detector (Fig. 1). The measurement cycle starts with a purging delay of two minutes followed by 10 separate measurements of one minute duration with a pause of 10 sec in between. The counted particles and size distributions are listed in Table 3. Each particle size distribution represents the median values of one measurement cycle. The impurities measured at the same time with the standard LINDE multi component detector are shown in Table 4.

TABLE 2. Main parameters of compressor and oil removal system.

	LINDE TCF50	**LINDE L700**	**LINDE L140**
Compressor	KAESER ESD 441 SFC	2x KAESER ESD 441	KAESER DSD 202 SFC
High pressure	12.5 bar @ 50 Hz	12 bar	7.8 bar @ 40Hz (part load)
Coalescers	1st and 2nd: DOMNICK HUNTER, AA-1300F/W, 57 l 3 filter elements: 340.TX.SAM.559 3rd: Upgrade August 2012 BEKO Clearpoint L102-S, 63 l 3 superfine filter elements 0.01 µm	3 x ROMABAU, 80 l	3 x KASAG DN125, 12.2 l
Charcoal adsorber	APARATEBAU AG, 340 l	APARATEBAU AG, 700 l	KASAG HAD 15-DN350, 156 l

TABLE 3. Particle size distribution (Median values of 10 measurement cycles).

	0.1 µm	0.15 µm	0.2 µm	0.25 µm	0.3 µm	0.5 µm	1.0 µm	5.0 µm
LINDE TCF50 (2 coalescer)	11809	1650	29	5	10	1	1	0
LINDE L700 (3 coalescer)	291	33	3	1	1	0	0	0
LINDE L140 (3 coalescer)	14	9	2	1	1	0	1	0
LINDE TCF50 (3 coalescer upgrade)	169	28	1	0	1	0	0	0

TABLE 4. Impurities measured with LINDE Multi Component Detector concurrently to particle measurements.

	H_2O [vpm]	N_2 [vpm]	Oil [ppb]
LINDE TCF50 (2 coalescer)	0.48	0.49	0.17
LINDE L700 (3 coalescer)	0.48	1.5	-2.43
LINDE L140 (3 coalescer)	0.3	0.1	0
LINDE TCF50 (3 coalescer upgrade)	0.38	0.48	0.62

In contrast to the particle numbers in Table 3, the impurities detected by the Multi Component Detector show no significant difference between the TCF50 with two coalescers and three coalescer systems. All values are near the lower detection limit level of the MCD. The negative value results from internal calculation and represents a measured oil concentration of 0 ppb.

The exceptionally high number of particles with a size of 0.1 µm and 0.15 µm at the LINDE TCF50 compared to the three coalescer systems caused us to install a third coalescer on this plant. Additionally we installed a bypass line and differential pressure transducers (KELLER PD23 / 0.5 bar) for further investigations on the oil removal system (ORS). In Fig. 5, the final installation is described. With this setup the pressure drop of each coalescer and of the charcoal adsorber can be determined. The observed pressure drops at compressor operation with 50 Hz and 12.5 bar were 167 mbar across the 1st coalescer, 51 mbar across the 2nd coalescer, 19 mbar across the 3rd coalescer, and 34 mbar across the charcoal adsorber. The comparably high pressure drop over the first coalescer is caused by the high oil saturation of the filter elements.

With the installation of a bypass line it is possible to switch from a three coalescer setup back to the original configuration. The ports for the particle measurements are located after second coalescer (A), after third coalescer (B), and after charcoal adsorber (C). With respect to the maximum concentration limit, and to avoid contamination of the particle counting system, measurements have not been taken upstream of the 2nd coalescer. The particle size distributions are listed in Table 5.

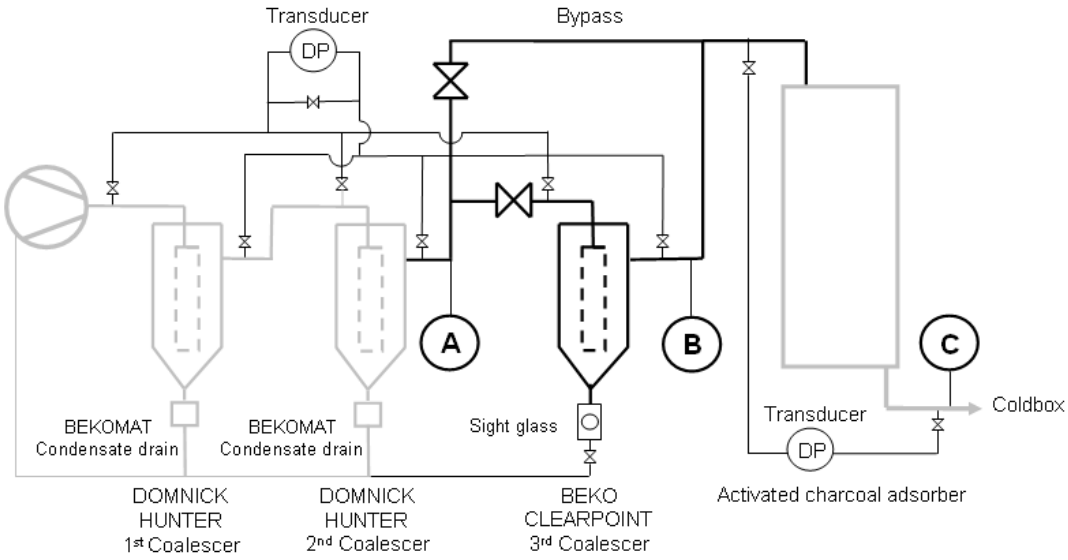


FIGURE 5. LINDE TCF50 Oil Removal System upgrade (standard configuration in grey colour).

TABLE 5. Particle size distribution after TCF50 ORS upgrade (Median values of 10 measurement cycles).

	0.1µm	**0.15 µm**	**0.2 µm**	**0.25 µm**	**0.3 µm**	**0.5 µm**	**1.0 µm**	**5.0 µm**
After coalescer 2 (A)	65353	8913	572	388	1201	190	37	10
After coalescer 3 (B)	1327	240	74	73	201	33	10	3
After charcoal adsorber (C) (3rd coalescer working)	208	32	3	1	1	0	0	0
After charcoal adsorber (C) (3rd coalescer bypassed)	11199	1559	34	7	20	3	0	0

The separation efficiency (E) of a filter is given by Eq.1 where N_{in} and N_{out} are the number of particles at the inlet and outlet of the filter. Figure 6 illustrates the calculated separation efficiencies for 3rd coalescer, the charcoal adsorber with bypassed 3rd coalescer, and the efficiency of 3rd coalescer and charcoal adsorber in combination. The number of counted particles at measuring point A after 2nd coalescer was the reference (N_{in}).

Whereas the separation efficiency of the 3rd coalescer with superfine filter grade decreases with the increasing size of the particles, the charcoal adsorber shows a worse efficiency at very small aerosol particles. An almost complete oil separation is achieved when a 3rd coalescer and charcoal adsorber are both enabled.

$$E = 1 - \frac{N_{out}}{N_{in}} \tag{1}$$

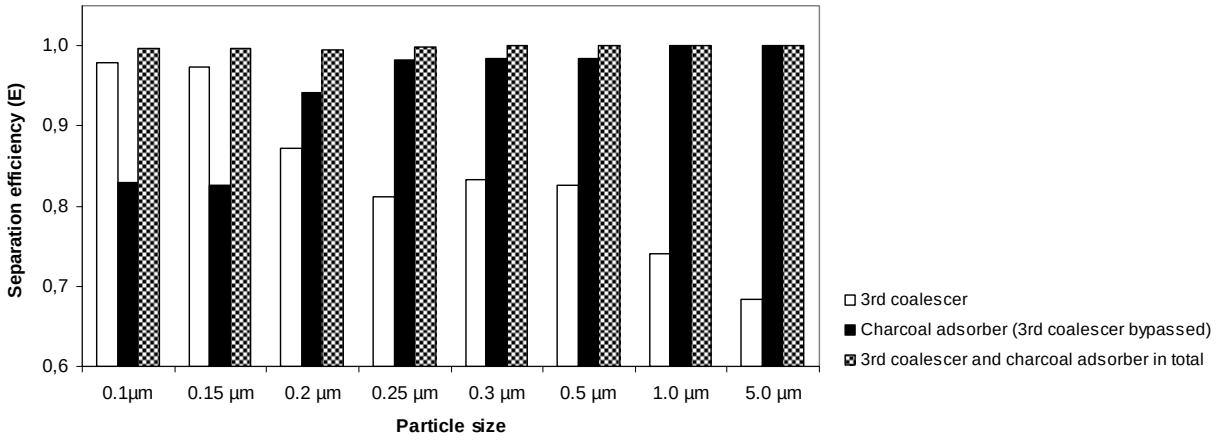


FIGURE 6. Separation efficiencies of the 3rd coalescer and charcoal adsorber for particle sizes 0.1 µm - 5 µm.

CONCLUSIONS

The described particle measurement configuration can be used as a diagnostic instrument to compare and evaluate the aerosol concentration on various helium plants. It is not suitable for permanent monitoring because of the permanent loss of gas through the HPD and the particle counter. A significant particle loss has to be assumed due to the fact that the particle counter as well as the HPD is used outside the manufacturer specifications as well as the sub-optimal gas sampling at a T-fitting and the transport through flexible tubes. A counting efficiency of only 50% at particle size of 0.1 µm increases this uncertainty. Therefore, a reliable statement about the absolute oil concentration in the high pressure line can not be made with the measured values. But, by using the same

measurement configuration on several sampling points as well as on different helium plants, the collected data can serve as a basis for the evaluation and development of oil removal systems.

In the case of the HZB LINDE TCF50 plant, the measurements have shown that the standard oil removal system was not sufficient to avoid the contamination of the coldbox. Compared to two other helium plants, each equipped with a three coalescer ORS, a large number of oil aerosol particles passed the two external coalescers and the final charcoal adsorber. An upgrade with an additional third coalescer reduced the residual oil concentration to the same level as on the reference plants.

The particle size distributions in Table 3 and Table 5 show that sub micron particles smaller than 0.15 µm represent the majority of particles passing through the oil removal system. It can be reasonably assumed that a high number of aerosol particles exist below the lower measuring limit of 0.1 µm. This very small particle size is probably the reason for the failing of the Multi Component Detector measurements at low oil concentrations. The upstream pyrolizer collects oil particles in a sinter filter for a certain period of time. Then the filter is heated to pyrolize the collected oil before the spectral analysis is done in the MCD [4]. If the particles are small enough to pass through the sinter filter, nothing can be detected in the MCD (Fig 1).

Coalescing filters with a superfine filter grade are the most efficient devices to remove very small aerosol particles from the helium gas. The charcoal adsorber shows a lower efficiency for the removal of small aerosol particles < 0.2 µm. An oil removal system consisting of three coalescers with a selection of appropriate filter grades together with a downstream charcoal adsorber is considered to be the optimal filter configuration.

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