LD8000-MULTIGAS

LDetek

CONFIGURED FOR CRYOGENIC HELIUM APPLICATION

DESIGN REPORT

▲ LD8000 MultiGas

▲ PlasmaDetek2

This technical document follows the application note LD16-06 on the use of LD8000-Multigas for the quality control of helium used in cryogenic stations.

The measurement of the trace impurities N2-O2-H2O-CnHm in a helium or argon matrix can be carried out with a PED type detector (PlasmaDetek2) US patent 9,310,308 B2 integrated into an instrument of the LD8000-Multigas type. This technology is based on cold micro-plasma excited at high voltage / frequency in a helium atmosphere and allowing the selective measurement of each component at a precise optical wavelength. The optical circuit is composed of photodiodes and interference filters combined with an amplification system for converting the measured photons into voltage. All signal processing is then redirected to a microcontroller.

Several variants and options on this device are possible depending on the needs of the customer. In this document, we will explain three types of assemblies possible for applications in a helium matrix.

Figure 1 **shows an installation where the required measurement must be configured for the following ranges (low to high ppm configuration):**

IMPURITIES	N_2	O_2	H_2O	CnHm
Range	0-300 ppm	0-50 ppm	0-100 ppm	0-30 ppm

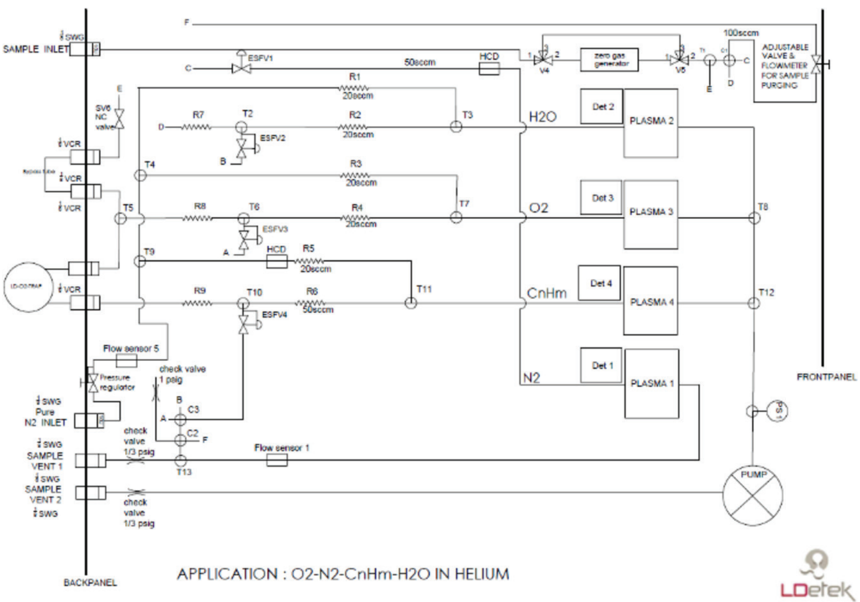

Figure 1

Figure 1 **demonstrates an instrument configured as follows:**

1. Four plasma detectors have been installed in the instrument for N2-O2-H2O-CnHm measurement. Each detector is configured with a narrow optical interference filter providing an optical response specific to the measured impurity. Each plasma is a monolithic structure made of quartz through which a pipe allows a linear gas flow. Through this linear flow, a scanning by an electric discharge is applied. This discharge is called “dielectric” because these electrodes are mounted externally of the quartz assembly. This ensures the inertia of the block internally being in contact with the gas. The discharge is said to be alternative to a specific high frequency and voltage.

2. The N2 doping input is required since the trace measurement of N2 can reach 300ppm. The N2 doping is then applied to the PEDs used for H2O-O2-CnHm measurement to eliminate the interference from the nitrogen beyond 10ppm. The nitrogen flow is generally controlled to the same value as the helium sample flow. We therefore speak of a ratio of 50% He / N2. Figure 1 shows a value of 20sccm helium for 20sccm nitrogen. Total nitrogen consumption for the instrument is therefore 60ml/min. The nitrogen flow rate is controlled by static orifices pre-adjusted to a defined pressure. To ensure the stability of the nitrogen flow, a mini pressure regulator is mounted on the instrument as well as a pressure flow sensor. This assembly ensures the stability of the nitrogen flow rate which is very important for maintaining the N2-He mixture equilibrium in the plasmas.

3. A vacuum pump is required at the PED output used with N2 / He mixture to allow the excitation of the PEDs performing the O2-H2O-CnHm measurement. The same pump is therefore common to the three PEDs. By this output, the measured flow is generally around 120 ml/min of a mixture of 50% He / N2. The 2nd output, this one without a vacuum pump, is specific to the PED for N2 measurement. The flow of helium on this output is 50ml/min from the PED for the measurement of N2, and 3 x 50ml / min being the bypass flow rates of the 3 sample regulators for the O2-H2O-CnHm PEDs. To these flow rates, is added the bypass flow at the inlet of the device serving to purge the line upstream of the instrument generally set at 100 ml/min. The total flow of clean helium that can therefore be recovered by the customer is around 300ml / min. Consequently, it is a total flow rate of 300ml / min + 60ml / min, i.e. 360ml / min of helium that is consumed. It is therefore possible to recover more than 83% of the helium consumed by the instrument. A system of “check valve” out of the PED was set up to recover helium without the risk of pressurization of the PEDs. The internal pressure PED is therefore limited to 1/3psig avoiding any risk of rupture of the quartz.

4. A zero-gas free system is installed to generate ultra high purity gas by flowing through a heated purifier and to do a zero calibration. It simply allows to use the sample gas of the customer and to purify it generating the 8.0 grade Helium (i.e., 99.999999%) having maximum impurity level of 10ppb. This purification allows to avoid a bad zero calibration and negative readings on the "process" gas of the customer. Moreover, there is no need for installation of an extra ultra-purity gas bottle with an external purification system, which is generally required by our competitors.

5. Each PED has its own electronic flow control by pressure flow regulator to ensure stability of reading regardless of the inlet pressure fluctuation. All flow controllers are "bypassed" to prevent contamination of the sample and ensure the best possible response time at a small flow rate. Generally, systems measuring N2-O2-H2O must operate at a high rate to allow a good sweep and dilution of the contamination to minimize drift and long response time. Thanks to our 'bypass' design, we can afford to keep small flows and limit the waste of Helium being generally very expensive.

6. An O2 trap with VCR connections is installed on the CnHm PED to eliminate oxygen interference on the CnHm measurement. This trap is composed of activated copper powder.

7. Humidity interference is removed by HCDs (H2O permeation tubes) installed on N2-CnHm PEDs. Providing a stable concentration of moisture to the N2-CnHm plasmas, they are saturated with water, removing the interference caused by moisture in the sample. If the presence of moisture in the sample is above 10-20ppm, then a second trap, this time to capture moisture, is required. It is also with VCR connections, consists of molecular sieve 3A. When it is not required, a simple ''bypass'' is mounted on the instrument.

8. A software protection system against carbon deposits in the PEDs allows them to go out when the CnHm measurement rises above 3ppm (adjustable parameter). By cutting the excitation of the N2-O2-H2O PEDs during high CnHm measurements, it is possible to prevent the degradation of the PEDs caused generally by the creation of carbon deposits on the walls of the monolithic quartz block.

9. In this configuration, by using the nitrogen doping for the O2 measurement, the nitrogen level is saturated and any form of interference from the N2 concentrations is avoided. In addition, the installation of optional moisture trap allows to eliminate any interference on O2 measurement from H2O concentrations. The O2 measurement shielded by its selective optical spectrum combined with N2 doping and H2O blocking by a trap can be used to compensate the N2 measurement. This compensation is done at the software level by an algorithm.

10. The tubing and fittings in contact with the line used for the H2O measurement are treated with a water-repellent inert coating to minimize surface absorption and provide better temperature stability and response time.

INTERFERENCE SUPPRESSION SYSTEM:

The protection system of the PEDs in the presence of too high hydrocarbon measurements, combined with O2-H2O trap networks and the humidity and nitrogen dosing system allows the LD8000-Multigas to remove interferences between the impurities. This is a unique LDetek practice, allowing accuracy and prolonged lifetime of the detector.

In order to protect the H2O and O2 traps, they are isolated by a '' shut off '' valve when the N2 measurement rises above 200ppm (adjustable parameter). The logic behind this protection is based on the fact that if the N2 measurement rises to a high level, then there is a strong possibility that it is an air infiltration and the same fact increases the O2-H2O concentrations. So, as long as the N2 measurement remains high, a protection preventing the sample from going to the O2-H2O traps remains activated.

Table of effects caused by interferents*:**

SAMPLE*	10 PPM N_2 O_2 CH_4 H_2O	10 PPM N_2	5 PPM N_2	10 PPM O_2	10 PPM N_2 O_2 CH_4
N_2	10 ppm	10 ppm	5 ppm	0 ppm	10 ppm
O_2	10 ppm	0 ppm	0 ppm	10 ppm	10 ppm
CnHm	10 ppm	0 ppm	0 ppm	0 ppm	10 ppm
H_2O	10 ppm	0 ppm	0 ppm	0 ppm	0 ppm

*The balance of the sample is helium.
*** Other combinations have been tested successfully, so have not been presented in this table. Only critical interference has been presented in this table.

MAINTENANCE:

Frequency 12-18 months:

Regeneration of O2 and H2O traps depending on the contents present in the sample.

Frequency 2-3 years:

Replacement of the vacuum pump.

Frequency 4-5 years:

Replacement of the HCD (permeation tube) normally performed at the same time as the replacement of the internal mini-purifier as well as the external compact purifier required for nitrogen dosing.

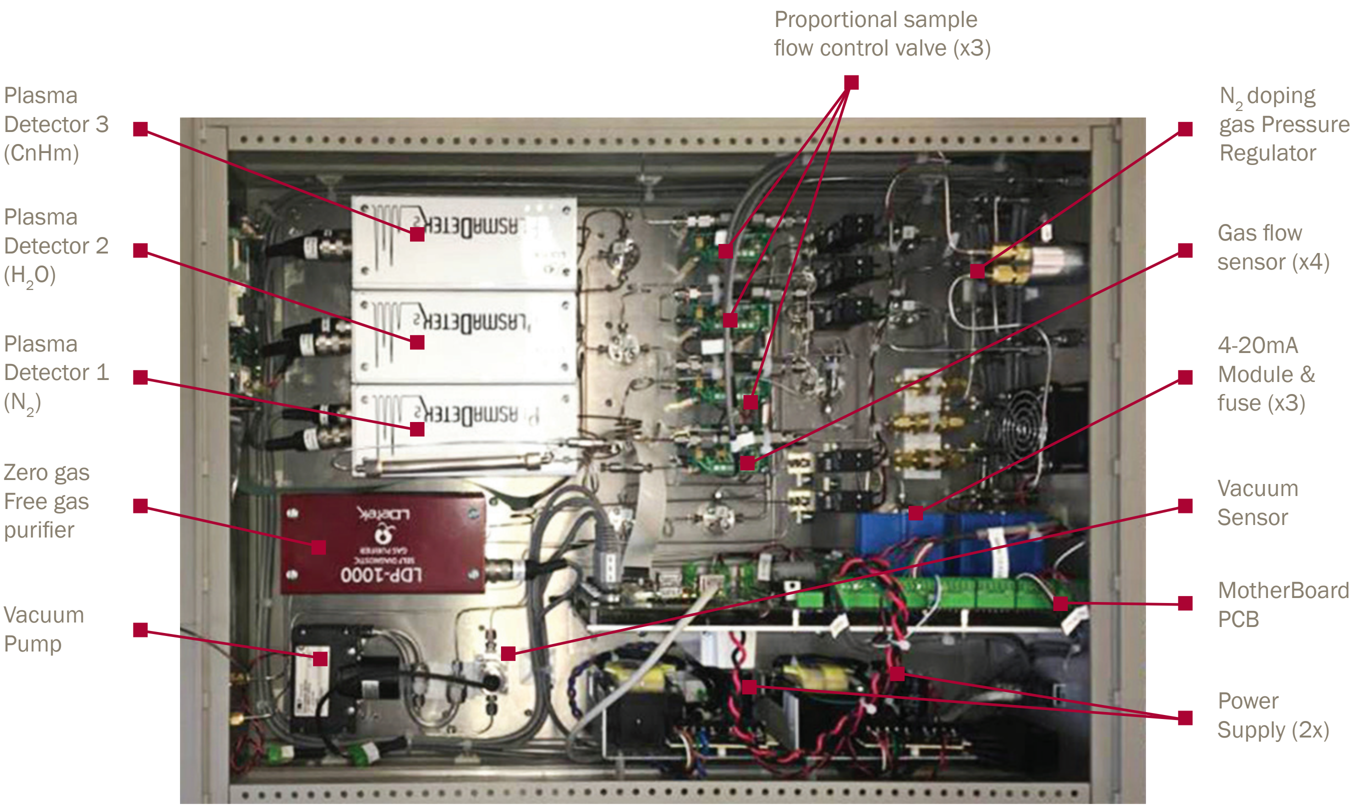

Photo 1

Photo of a configuration based on Figure 1 but for N2-CnHm-H2O measurements without O2 measurement. Other configurations are based on the same mounting but with slightly different module layouts to accommodate the same 4U rack mount cabinet.

Conclusion on the configuration of Figure 1:

This is the most complete configuration of all. It shows no interference between the components to be measured as shown in the table. In addition, this configuration can cover the full range from low ppb / ppm to higher ppm. It is therefore the most robust, stable and precise solution for this type of application.

Figure 2 **shows an installation where the required measurement must be configured for the following ranges (low ppm configuration only):**

IMPURITIES	N_2	O_2	H_2O	CnHm
Range	0-10 ppm	0-10 ppm	0-10 ppm	0-10 ppm

Figure 2

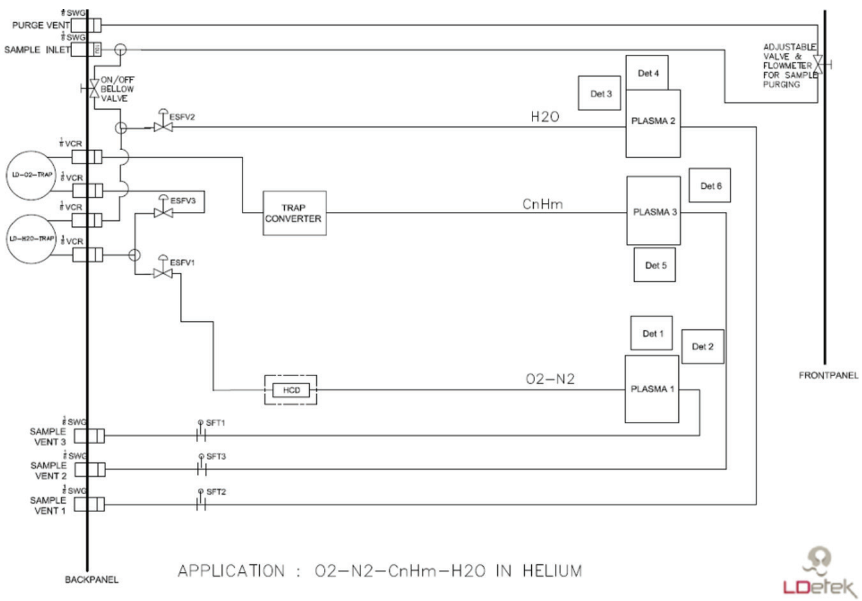

Figure 2 **demonstrates an instrument configured as follows:**

1. Three plasma detectors have been installed in the instrument, respectively for the N2 / O2-H2O-CnHm measurement. Each detector is configured with a narrow interference optical filter providing an optical response specific to the measured impurity. Each plasma is a monolithic structure made of quartz through which a pipe allows a linear gas flow. Through this linear flow, a scanning by an electric discharge is applied. This discharge is called “dielectric” because these electrodes are mounted externally of the quartz assembly. This ensures the inertia of the block internally being in contact with the gas. The discharge is said to be alternative to a specific high frequency and voltage.

2. Nitrogen doping is not required because the N2 range is below 10ppm and the interference caused by nitrogen on O2-CnHm-H2O measurements is limited.

3. A vacuum pump is not required at the PED output for the same reason as indicated in point 1.

4. Each PED has its own electronic flow control regulated by a proportional microvalve with minimal internal volume. The reading of the flow is made by mass sensors installed at the outlet of the PEDs to avoid any form of contamination and additional volume in front of the detector. Our design allows the use of small flow since no bulky flow control element is installed upstream of the detectors. All tubing is made of 1 / 16’’OD stainless steel to minimize purge volumes. A water-repellent “coating” is applied for the plasma pipes used for H2O measurement to reduce surface absorption. The flow rates at the outlet of each PED are respectively 50 ml / min for a total of 150 ml / min. If this flow rate is combined with the flow rate at the inlet, which is generally adjusted to 100 ml / min, the total flow rate of the instrument is thus 250 ml / min. All this flow can be recovered because it has not been contaminated by the instrument.

5. Moisture interference is removed by installing HCD (H2O permeation tube) on PED O2 / N2. Providing a stable concentration of moisture to the O2 / N2 plasma, it is saturated with water, removing the interference caused by moisture in the sample. If the presence of moisture in the sample is above 5-10ppm, then a second trap, this time to capture moisture is required. It consists of molecular sieve 3A and has VCR fittings. When it is not required, a simple ‘’ bypass ‘’ is mounted on the instrument.

6. A software protection system against carbon deposits at the PEDs allows them to go out when the CnHm measurement rises above 3ppm (adjustable parameter). By cutting the excitation of the N2 / O2-H2O PEDs during high CnHm measurements, it is possible to prevent the degradation of the PEDs generally caused by the creation of carbon deposits on the walls of the monolithic quartz block.

7. In this configuration where the plasma nitrogen dosage is not used, it should not be surprising that O2-H2O traps should be used at a lower concentration to eliminate interference than we see in a platform where the nitrogen dosage is in place. This phenomenon is caused by the equilibrium of the species inside the plasma, offering a different mode of operation. It is also for the same reason that in this configuration under Helium plasma only, we use a system of "trap converter" which consists of a mini plasma allowing the carbon to decompose thus allowing the PED CnHm to measure a '' by-product '' from the carbon decomposition. This allows a more selective measurement of hydrocarbons and at the same time avoids the CnHm PED to make carbon deposits on these walls since the carbons remain in the space of the trap converter system protecting the plasma used to measure the CnHm.

8. In this configuration, each PED contains an additional optical circuit, selective to the N2 measurement. This allows leak diagnosis in each channel.

INTERFERENCE SUPPRESSION SYSTEM:

The protection system of the PEDs in the presence of too high hydrocarbon measurements, combined with O2-H2O trap networks, moisture metering system and carbon converter allows the LD8000-Multigas to minimize the interferences between the impurities. This is a unique LDetek practice, allowing accuracy and prolonged lifetime of the detector.

Table of effects caused by interferents*:**

SAMPLE*	10 PPM N_2 O_2 CH_4 H_2O	10 PPM N_2	5 PPM N_2	4 PPM O_2	10 PPM N_2 O_2 CH_4
N_2	10 ppm	10 ppm	5 ppm	-2 ppm** (display = 0 ppm)	10 ppm
O_2	10 ppm	-3 ppm** (display = 0 ppm)	-2 ppm** (display = 0 ppm)	4 ppm	10 ppm
CnHm	10 ppm	0 ppm	0 ppm	0 ppm	10 ppm
H_2O	10 ppm	+0.2 ppm**	+0.1 ppm**	+0.2 ppm**	+0.5 ppm**

* The sample balance is helium
** Approximate measurement based on laboratory tests
*** Other combinations have been successfully tested, so they are not presented in this table. Only critical interference is shown in this table.

MAINTENANCE:

Frequency 12-18 months:
Regeneration of the O2 and H2O traps according to the contents present in the sample.

Frequency 4-5 years:
HCD (permeation tube) replacement.

Conclusion on the configuration of Figure 2:

This is the simplest and cheapest configuration of all, but it presents some interference between the compounds to be measured as shown in the table. On the other hand, these interferences can be negligible according to the needs and the application of the customers.

Figure 3 shows an installation where the required measurement must be configured for the following ranges (so-called low to medium ppm configuration):

IMPURITIES	N_2	O_2	H_2O	CnHm
Range	0-20 ppm	0-20 ppm	0-20 ppm	0-20 ppm

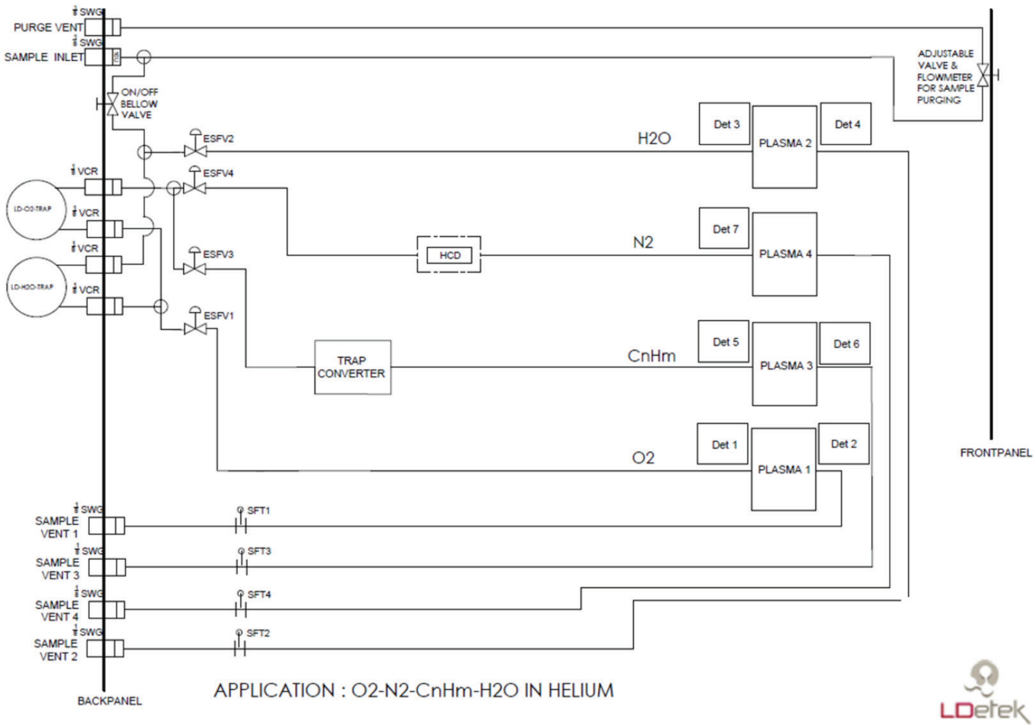

Figure 3

Figure 3 demonstrates an instrument configured as follows:

1. This configuration is similar to the one described in Figure 2. However, it allows to cover a so-called average measurement range, i.e. to go up to 20ppm ranges. To enable this, a plasma specifically configured for N2 measurement has been added behind the H2O and O2 traps allowing to obtain a measurement without interference of nitrogen. In this configuration, the shielded nitrogen measurement is used to apply compensation to the O2 measurement.

INTERFERENCE SUPPRESSION SYSTEM:

The protection system for PEDs in the presence of excessively high hydrocarbon measurements, combined with O2-H2O trap networks, moisture metering system and carbon converter allows the LD8000-Multigas to eliminate interference between the impurities. This is a unique LDetek practice, allowing accuracy and prolonged lifetime of the detector.

Table of effects caused by interferents*:**

SAMPLE*	10 PPM N_2 O_2 CH_4 H_2O	10 PPM N_2	5 PPM N_2	10 PPM O_2	10 PPM N_2 O_2 CH_4
N_2	10 ppm	10 ppm	5 ppm	0 ppm	10 ppm
O_2	10 ppm	0 ppm	0 ppm	10 ppm	10 ppm
CnHm	10 ppm	0 ppm	0 ppm	0 ppm	10 ppm
H_2O	10 ppm	+0.2 ppm**	+0.1 ppm**	+0.2 ppm**	+0.5 ppm**

* The sample balance is helium
** Approximate measurement based on laboratory tests
*** Other combinations have been successfully tested, so they are not presented in this table. Only critical interference is shown in this table.

MAINTENANCE:

Frequency 12-18 months:

Regeneration of the O2 and H2O traps according to the contents present in the sample.

Frequency 4-5 years:

HCD (permeation tube) replacement.

Conclusion on the configuration of Figure 3:

This is the configuration that lies between the simplest and most complete configurations previously explained (Figures 1 and 2). It allows a little higher ranges measurement and interference minimization without the use of nitrogen doping. Depending on the needs and application, this configuration can be considered advantageous and is a good compromise between the two extremes.