LD8000

LDetek

TRACE NITROGEN IN ARGON,
HELIUM AND CRUDE ARGON ANALYZER

. . . specifically for an ***On-line Analyser***
.> pertinent to Gas Industry
O2Sep Plants / Steel making

APPLICATION NOTE
LD19-05

LDetek

Measurement of impurities in UHP Argon for HIP process
using the MultiDetek2 and PlasmaDetek2

more details specifically
> re HIP Steel making

LD8000 MULTIGAS

LDetek

TRACE NITROGEN, OXYGEN, MOISTURE, HYDROCARBONS
IN HELIUM

Trace Gases ONLY !
> an extended process GC
. . . but potential for a <u>decent</u>
"Lab" GC Instrument
with some more "imagination !

APPLICATION NOTE
LD19-05

Measurement of impurities in UHP Argon for HIP process using the MultiDetek2 and PlasmaDetek2

▲ MultiDetek2

▲ PlasmaDetek2
patent US 9,310,308 B2

Argon is a widely used gas in different needs such as steel industries, air separation, welding, purging, chemical plants, semiconductor and others. Having a good analytical tool is mandatory to ensure the required purity of argon.

This application note is the continuity of the LD16-07. In this application note, we will particularly covers the HIP (hot isostatic pressing) process in the steel industry with the use of the MultiDetek2 gas analyser GC for trace impurities in Argon.

What is the HIP?

HIP combines very high temperatures, very high pressures, and inert gas to eliminate porosity in castings and consolidate powder into dense materials. Temperatures are up to 2,000 deg C, pressures are up to 30,000 psi and UHP inert gas generally argon are the conditions required.

What is the utility of using HIP?

The hot isostatic pressing is used extensively to prolong the working life of components and protect them from environmental factors such as corrosion and abrasion. It provides thermal processing which improve material properties such as strength, durability and corrosion resistance, enabling manufacturers' components to work more efficiently with significantly extended operational lifetimes. It is used to alter the microstructure of materials, such as metals and alloys, to impart properties which benefit the working life of a component, for example: increased surface hardness, temperature resistance, ductility and strength. Hot isostatic pressing uses very high pressures in addition to high temperature to achieve engineering outcomes that are impossible by other methods. HIP is used to eliminate porosity in castings and consolidate encapsulated powders to dense materials. Dissimilar materials can be bonded together to manufacture unique, cost effective components.

What are the markets that require products to be manufactured from this HIP process?

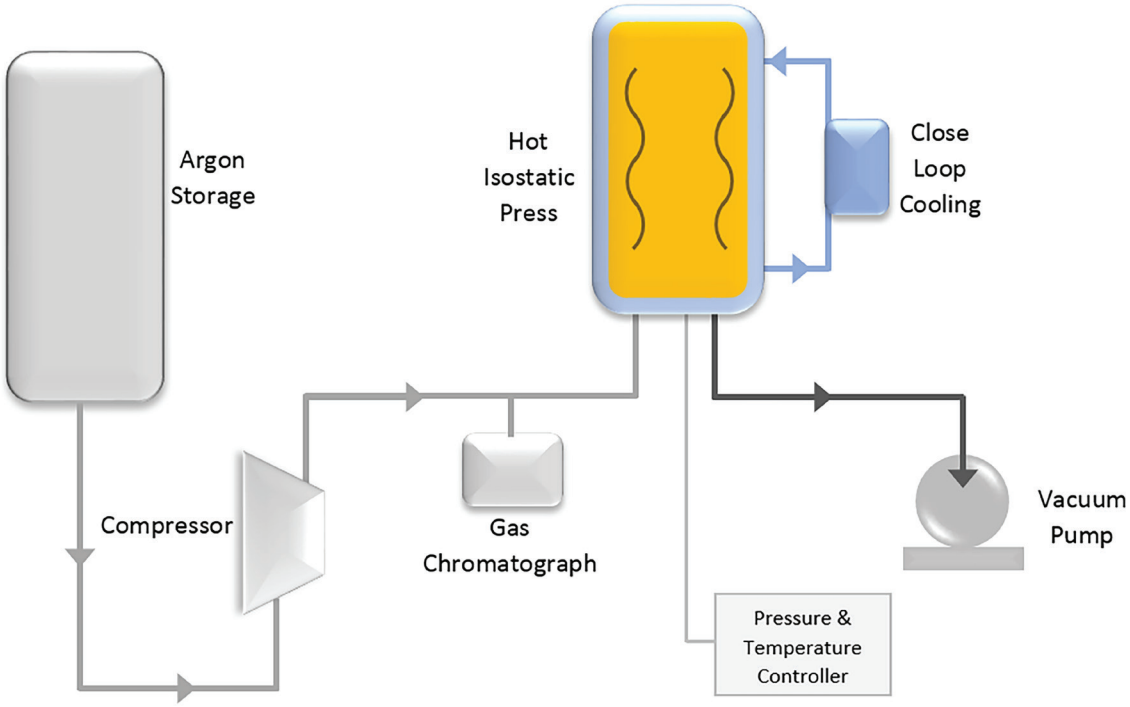
HIP installations will process many tons of titanium, aluminium, steel and super-alloy castings, removing porosity and improving the performance of parts such as turbine blades and oilfield components. In almost all cases metals in any state require heat treatment to improve their properties, if components are to achieve desired levels of longevity and corrosion performance in use. Without heat treatment car engine components, for example, might last for hundreds of miles rather than the tens of thousands we expect. The use of the specialist surface technologies offers further improve in-use characteristics, particularly in severe environments such as in aircraft engines and in sub-sea oil & gas applications.

Why gas analysis is required for HIP?

An inert gas is required to prevent chemical reactions during the HIP process at elevated temperatures, making argon the ideal candidate. Monitoring of the HIP argon gas and its impurities is required to control the quality and repeatability of the HIP process.

The general quality control requires the analysis of the trace impurities in a range of 0-100ppm for H2-O2-N2-CH4-CO-CO2-NMHC-H2O in UHP argon.

HIP typical installation:



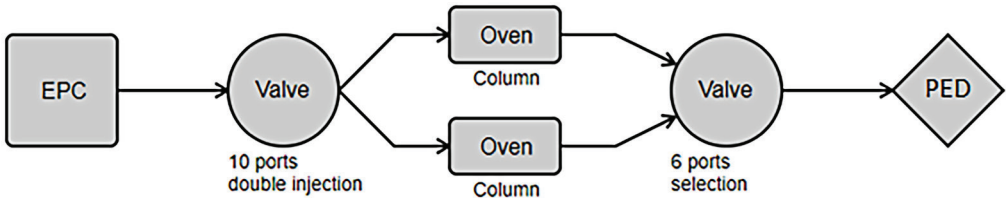
LDETEK SOLUTION:

The MultiDetek 2 combined with the PlasmaDetek 2 detector provides an ideal solution to measure the different impurities in UHP Argon.

The system is simply configured with 2 blocks, each one merging in one PED. Each block has its own chromatography column mounted in a compact isothermal oven. A simple injection with sampling loop technique mounted on a diaphragm valve is used to introduce the sample gas to the detector.

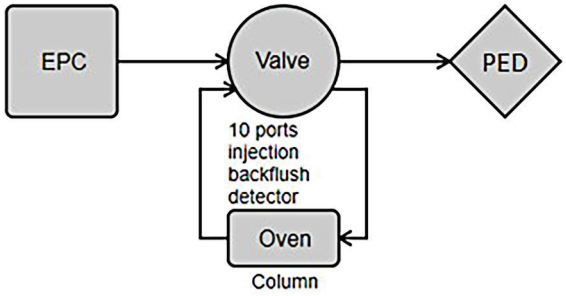
Block 1 is used to measure trace H2-O2-N2-CH4-CO and CO2

For block 1 configuration, a selection diaphragm valve is used for synchronizing the impurities coming out of the columns to the plasma detector. The PED is configured with selective optics for each measured impurity improving the sensitivity.



Block 2 is used to measure trace NMHC

The block 2 arrangement allows to inject light impurities to vent and by reverting the valve position, the C2s-C3s-C4s hydrocarbons are grouped together as one peak to form NMHC to the PED. The PED has the right optic, selective to hydrocarbons.

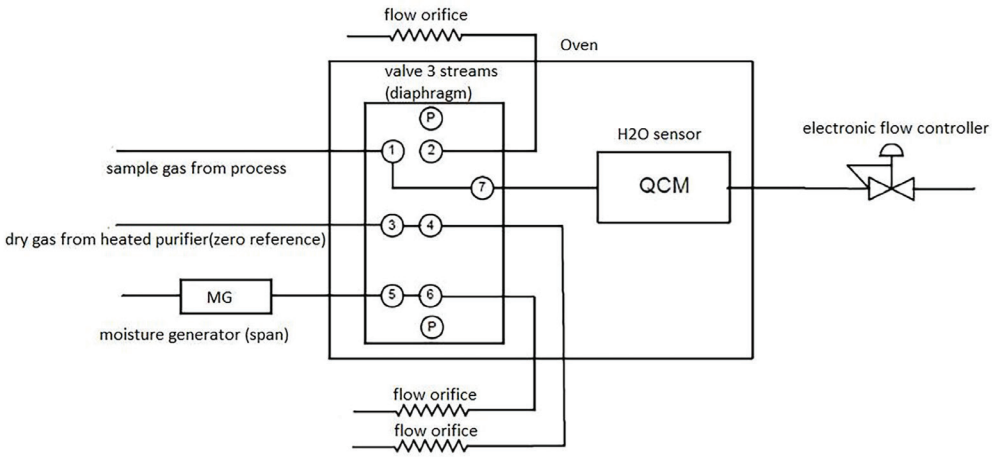


For the analysis of trace H2O, 2 solutions are offered depending of the requirements.

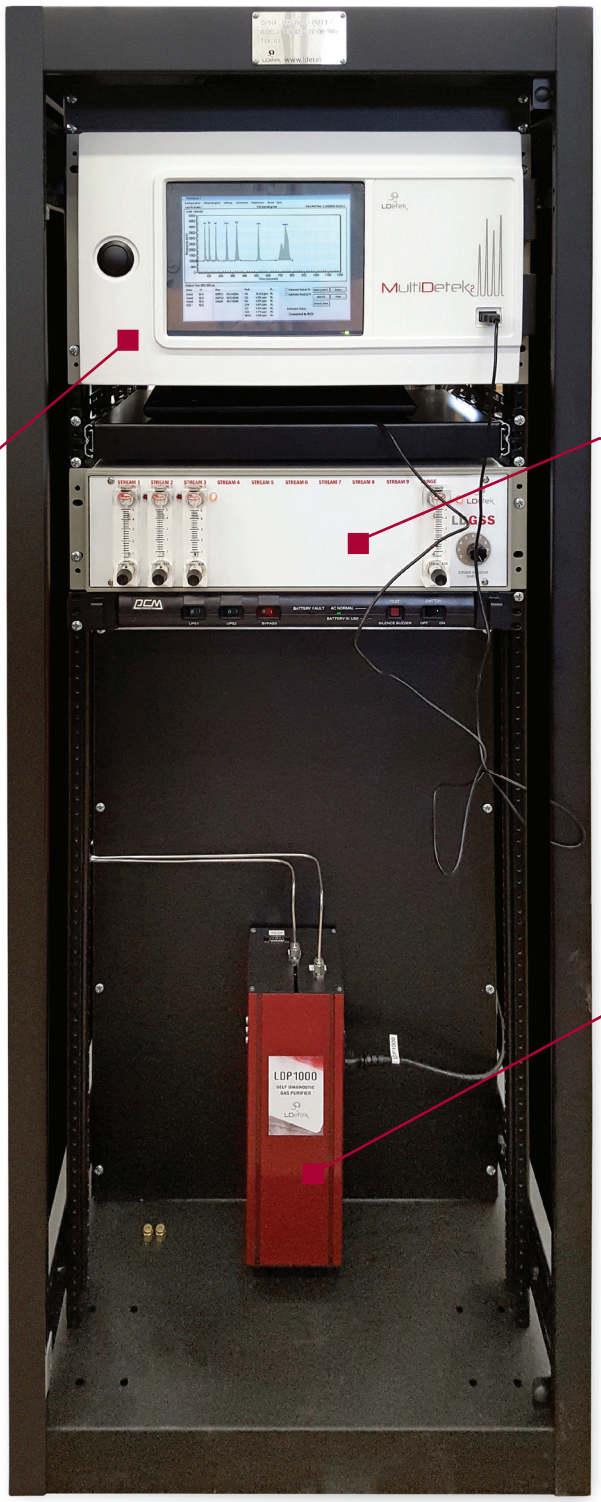
1. A Michell DewPoint sensor can be added to the MultiDetek2, connected in parallel. The 4-20mA output of the sensor is wired back to the MD2 analog input. Then, all results can be report on the GC interface. Going this way a LDL of 0.5ppm can be achieved for H2O impurity.

2. If an ldl lower than 0.5ppm is required, then a third block is mounted inside the MultiDetek2 containing a quartz crystal micro balance moisture sensor. Using this way, a span and zero calibration system is integrated inside the GC unit for calibrating the H2O sensor.

Block 3 is represented for measuring trace H2O



LDRACK INTEGRATED COMPLETE **SOLUTION**



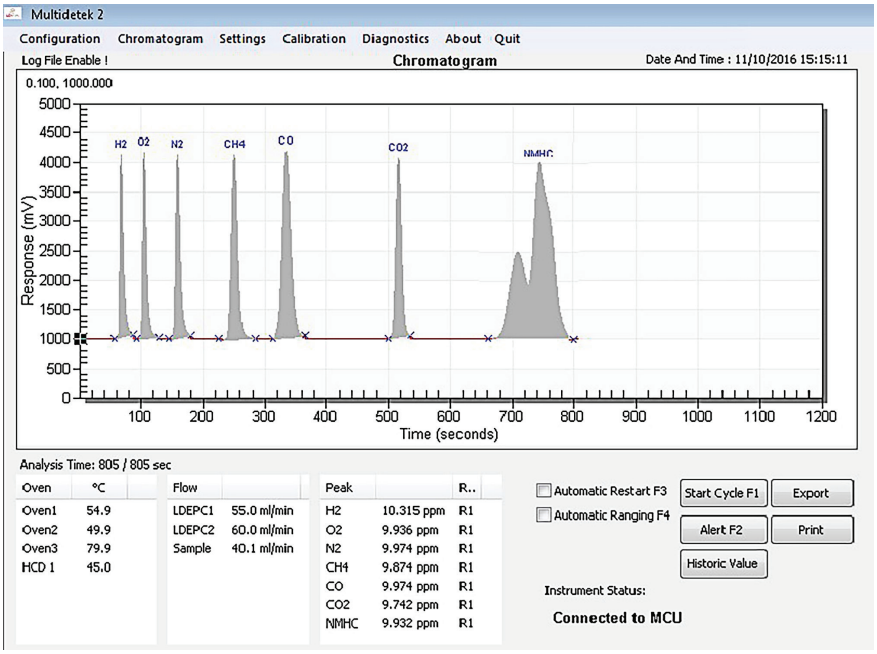
MULTIDETEK2
GC gas analyser for measuring trace H2-02-N2-CH4-CO-CO2-H20 in UHP argon.

LDGSS
ultra high purity gas stream selector system for switching between the different streams and the span calibration gas (Up to 10 streams in one device is available).

LDP1000
heated gas purifier to generate UHP grade 99.999999% from grade 99.999% for carrier gas of the GC.

RESULTS:

A chromatogram of such system with a standard gas containing trace impurities in a balance of Argon. An analysis time of less than 10 minutes is required for H2-O2-N2-CH4-CO-CO2 and by adding the NMHC impurity, the analysis time goes to 12 minutes.



The following chart gives the limit of detection for such GC configuration

COMPONENT	CONCENTRATION	PEAK HEIGHT	NOISE	LDL (3X NOISE)
H2	10.315 ppm	3202 mV	2.4 mV	0.023 ppm
O2	9.936 ppm	3221 mV	2.1 mV	0.019 ppm
N2	9.974 ppm	3205 mV	1.0 mV	0.010 ppm
CH4	9.874 ppm	3201 mV	2.0 mV	0.019 ppm
CO	9.974 ppm	3251 mV	2.6 mV	0.024 ppm
CO2	9.742 ppm	3191 mV	2.3 mV	0.021 ppm
NMHC	9.932 ppm	3051 mV	2.1 mV	0.021 ppm

Figure 2 Note: other LDL could be obtained with different injection volume and chromatographic condition

CONCLUSION:

With the MultiDetek2 compact GC combined with PlasmaDetek2 detector, the analysis of trace impurities in UHP argon can be realized in one rackmount instrument with one type of detector. The use of argon as carrier gas entails a low cost of operation. On top of that, the MultiDetek2 offers all the features and industrial protocols/controls required by the industrial market for such type of application.

Our fully integrated solution LDrack combining the analytical instrument MultiDetek2 with our stream selector system LDGSS with the integrated analysis for trace H2O makes a reliable turnkey solution for the HIP furnace manufacturers, 3D printer manufacturers and some controlled atmosphere manufacturers.

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APPLICATION NOTE
LD16-07

Measurement of impurities in UHP Argon using the MultiDetek 2 and PlasmaDetek 2

▲ MultiDetek2 ▲ PlasmaDetek2

Argon is a widely used gas in different needs such as steel industries, air separation, welding, purging, chemical plants, semiconductor and others. Having a good analytical tool is mandatory to ensure the required purity of argon.

The most popular technique for UHP argon analysis is to detect trace impurities by gas chromatography. Some of the most common technologies will use a combination of multiple detectors to achieve the analysis requirements. Most commonly used are FID (flame ionization detector) combined with PDD (pulse discharge detector). This technique requires the need of helium as carrier gas what is an expensive gas to be used as carrier gas for the analysis of H2-N2-CO-CO2. The analyses of hydrocarbons will be performed using the FID what requires extra cost due to air and fuel. On top of that, the oxygen analysis must be performed using a separated trace oxygen analyzer due to the co elution of argon and oxygen in the gas chromatography system with helium ionization detection technique.

LDETEK SOLUTION:

The MultiDetek 2 combined with the PlasmaDetek 2 detector provides an ideal solution to measure the different impurities in UHP argon. With the PlasmaDetek 2, based on plasma emission detection, impurities in low ppb can be easily detected.

The system is simply configured with 3 channels and one plasma detector. Each channel has its own chromatography column mounted in a compact isothermal oven. A simple injection with sampling loop technique mounted on a diaphragm valve is used to introduce the sample gas to the detector.

Channel 1 to trace H2-O2-N2-CH4-CO
Channel 2 to trace CO2
Channel 3 to trace NMHC (NMHC can be measured as required hydrocarbon equivalent depending on the need)

A diaphragm valve network is used for synchronizing the impurities to the plasma detector.

Figure 1 shows a chromatogram of such system with a standard gas containing trace impurities in a balance of Argon. Figure 2 shows the LDL that such Argon purity system can achieve based on noise level to signal ratio calculation.

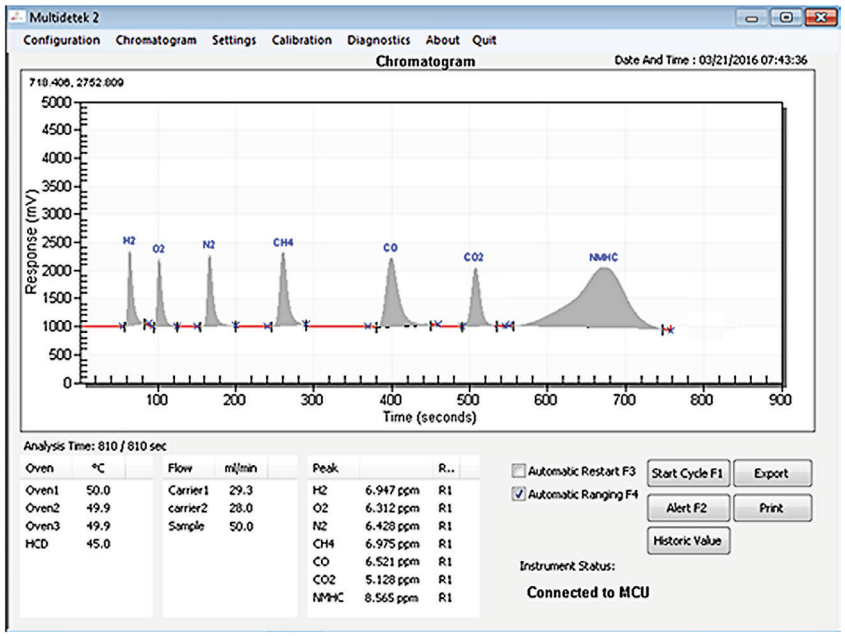

Figure 1

COMPONENT	CONCENTRATION	PEAK HEIGHT	NOISE	LDL (3X NOISE)
H_2	6.947 ppm	1391 mV	2.5 mV	0.037 ppm
O_2	6.312 ppm	1311 mV	2.1 mV	0.030 ppm
N_2	6.428 ppm	1377 mV	1.0 mV	0.014 ppm
CH_4	6.975 ppm	1390 mV	2.0 mV	0.030 ppm
CO	6.521 ppm	1270 mV	2.6 mV	0.040 ppm
CO_2	5.128 ppm	1168 mV	2.3 mV	0.030 ppm
NMHC	8.565 ppm	1201 mV	1.6 mV	0.034 ppm

Note: other LDL could be obtained with different injection volume and chromatographic condition

Figure 2

CONCLUSION:

Using the MultiDetek 2 compact GC, it becomes the most convenient solution for argon purity analysis. It is a maintenance free system that offers the required performances. The use of argon as carrier gas entails a low cost of operation. On top of that, the MultiDetek2 offers all the features required by the industrial market for such type of application.

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LD8000

TRACE NITROGEN IN ARGON,
HELIUM AND CRUDE ARGON ANALYZER

The LD8000 is an online analyzer to monitor trace N2 in Ar/He/Crude Argon. Plasma emission detector is used to selectively measure trace N2 in Argon or/and Helium bulk gases. The analysis of trace N2 in Neon, Xenon and Krypton can also be realized.

Plasma Emission Detector for N_2

FEATURES:

- Trace Nitrogen in Argon/Helium/Crude Argon
- Compact 3U rackmount enclosure
- Large scale measurement
- 4-20 mA outputs as standard
- Range Identification Relay
- Micro-valve for very low dead volume and fast purging time
- Low sample consumption
- Optional zero gas calibration free system

APPLICATIONS:

- Air separation unit
- Helium cryogenic installation
- Cryogenic truck loading station
- Speciality gas laboratories
- Process control
- Argon purification plant
- Steel Industries
- Chemical plants
- Welding gas control
- Helium liquification plants
- Gas management system
- Semiconductor manufacturing
- Quality control for truck fills and gas cylinders
- Inert glove box systems
- Universities and laboratories

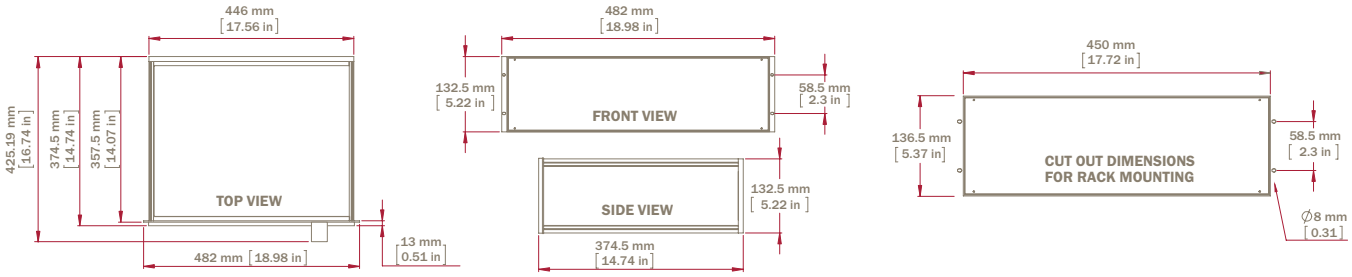
SPECIFICATIONS:

DETECTOR TYPES	Plasma Emission Detector for N_2	
RANGE FOR N_2	0 – 1 ppm, resolution to 10 ppb 0 – 10 ppm, resolution to .1 ppm	0 – 100 ppm, resolution to 1 ppm other range possible up to 5000 ppm configurable
STANDARD FEATURES	• Manual or autoranging (user selectable) • Microprocessor controlled • 5.6" TFT intelligent LCD module with Touch Screen • Self diagnosis system with auto-resolve alarm • 4-20 mA isolated outputs	• Alarm Historic • Safe calibration procedure to avoid any bad calibration • Digital ouputs for remote monitoring: (all dry relay contacts) - System status (1 output) - Range in use (3 outputs per impurity) - Calibration in use (1 output)
OPTIONS	• Internal sampling system for zero, span and sample	• Serial port: RS-232 / 422 / 485 / Profibus • 2 alarm outputs (user programmable set point) • Zero calibration gas free system
GAS CONNECTIONS	Sample: 1/8'' compression fittings	Vent: 1/8'' compression fitting
CALIBRATION GAS	Zero: LDP1000 purified gas (Getter)	Span: 8.0 to 9.5 ppm N_2 (application dependant)
SAMPLE FLOW REQUIREMENTS	75 to 200 sccm	
OPERATING TEMPERATURE	10 °C to 45 °C	
SUPPLY	115 VAC, 50 – 60 Hz or 220 VAC, 50 – 60 Hz	
ACCURACY	Better than ± 1% full scale	
DRIFT	< ± 1%	
RESPONSE TIME	T90 < 10 seconds	
OPERATING SAMPLE PRESSURE RANGE	3-30psig (for lower sample pressure requirement, an additional high purity pump is used)	
OUTLET PRESSURE	Atmospheric	
ENCLOSURE TYPE	3U rackmount type	
INGRESS PROTECTION	IP20 in accordance with IEC 60529	
ENCLOSURE FINISH	RAL7030 powder coat	
CERTIFICATION	In compliance with EMC directive 2004/108/EC, EN 61000-6-2:2005 for immunity & EN 61000-6-4:2007 for emissions	
WEIGHT	29 lbs (13 kg)	

ORDERING INFORMATION:

LD8000	-X	-X	-XXX	-X	-XX	-X	-XXX	-X
	N2: Nitrogen	**A:** Argon **H:** Helium **C:** Crude Argon **D:** Dual (Argon + Helium)	Operating Voltage: **120:** 120 volts **220:** 220 volts	**A:** Alarm option	Integrated sampling system **S1:** 1 sample + zero + span **S2:** 2 samples + zero + span	**C:** Zero gas free system	Serial communication: **RS2:** RS-232 **RS4:** RS-485 **PFB:** Profibus	**P:** Purge value and flowmeter

DIMENSIONS:



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LD8000 MULTIGAS

TRACE NITROGEN, OXYGEN, MOISTURE, HYDROCARBONS IN HELIUM

The LD8000 MultiGas is an online gas analyzer to monitor trace N2, O2, H2O, CnHm in Helium. The compact 4U unit has been designed to meet the requirements of the Helium cryogenic installations for online measurement.

FEATURES:

- Unique compact chassis (4U rack mount) to cover up to four measurements (N2, O2, H2O, CnHm)
- Bootloader integrated for software update via Ethernet
- Large scale measurement
- 4-20 mA outputs as standard
- Optional zero gas calibration free system
- Range Identification Relay
- LAN/Web control
- Micro-valve for very low dead volume and fast purging time
- Low sample consumption
- Front adjustment valve for sample bypass flow to purge the sample gas line before the analyzer

APPLICATIONS:

- Helium cryogenic installation
- Cryogenic truck loading station
- Process control
- Helium liquification plants
- Research center

SPECIFICATIONS:

DETECTOR TYPES	Plasma Emission Detector for N_2 - O_2 - H_2O - CnHm	
RANGE FOR N_2	0 – 10 ppm, resolution to 100 ppb	0 – 100 ppm, resolution to 1 ppm
RANGE FOR O_2	0 – 3 ppm, resolution to 100 ppb	0 – 30 ppm, resolution to .1 ppm
RANGE FOR H_2O	0 – 3 ppm, resolution to 100 ppb	0 – 30 ppm, resolution to .1 ppm
RANGE FOR CNHM	0 – 3 ppm, resolution to 100 ppb	0 – 30 ppm, resolution to .1 ppm
STANDARD FEATURES	• Manual or autoranging (user selectable) • Microprocessor controlled • 5.6" TFT intelligent LCD module with Touch Screen • Self diagnosis system with auto-resolve alarm • LAN/Web control	• 4-20 mA isolated outputs (one for each component) • Alarm Historic • Safe calibration procedure to avoid any bad calibration • Digital ouputs for remote monitoring: (all dry relay contacts) - System status (1 output) - Range in use (1 output)
OPTIONS	• Internal sampling system for zero, span and sample • Zero calibration gas free system	• Serial port: RS-232 / 422 / 485 / Profibus • 2 alarm outputs (user programmable set point)
GAS CONNECTIONS	Sample: 1/8'' compression fittings	Vent: 1/8'' compression fitting
CALIBRATION GAS	Zero: LDP1000 purified gas or Zero calibration gas free system Span 1: 8.0 ppm N_2 and O_2 + 3 ppm CH4 in Helium (application dependent)	Span 2: 8.0 ppm H2O in Helium (application dependent)
SAMPLE FLOW REQUIREMENTS	50 sccm for N2 channel (PED#1) 20 sccm for O2 channel (PED#2) 20 sccm for H2O channel (PED#3)	20 sccm for CnHm channel (PED#4) Bypass flow per channel : 100 sccm
NITROGEN DOPING GAS FLOW REQUIREMENT	20 sccm for O2 channel (PED#2) 20 sccm for H2O channel (PED#3)	20 sccm for CnHm channel (PED#4)
RECOMMENDED MINIMUM OPERATING PRESSURE	4 PSIG (28 kPAG) optional 1 PSIG (7 kPAG)	
OPERATING TEMPERATURE	10 °C to 45 °C	
SUPPLY	115 VAC, 50 – 60 Hz or 220 VAC, 50 – 60 Hz	
ACCURACY	Better than ± 1% full scale	
DRIFT	< ± 1% over 24 hours	
RESPONSE TIME	T90 < 60 seconds	
WEIGHT	45-60 lbs (20-27 kg) (depending of options)	

ORDERING INFORMATION:

LD8000 MULTIGAS	-X-X-X-X	-XXX	-X	-XXX	-X	-XXX
	N2: Nitrogen **O2:** Oxygen **H2O:** moisture **CnHm :** hydrocarbons	Operating Voltage: **120:** 120 volts **220:** 220 volts	**A:** Alarm option	Integrated sampling system **S1:** 1 sample + zero + span **S2:** 2 samples + zero + span	**C:** Zero gas free system	Serial communication **RS2:** RS-232 **RS4:** RS-485 **PFB:** Profibus

DIMENSIONS: