

# 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?**

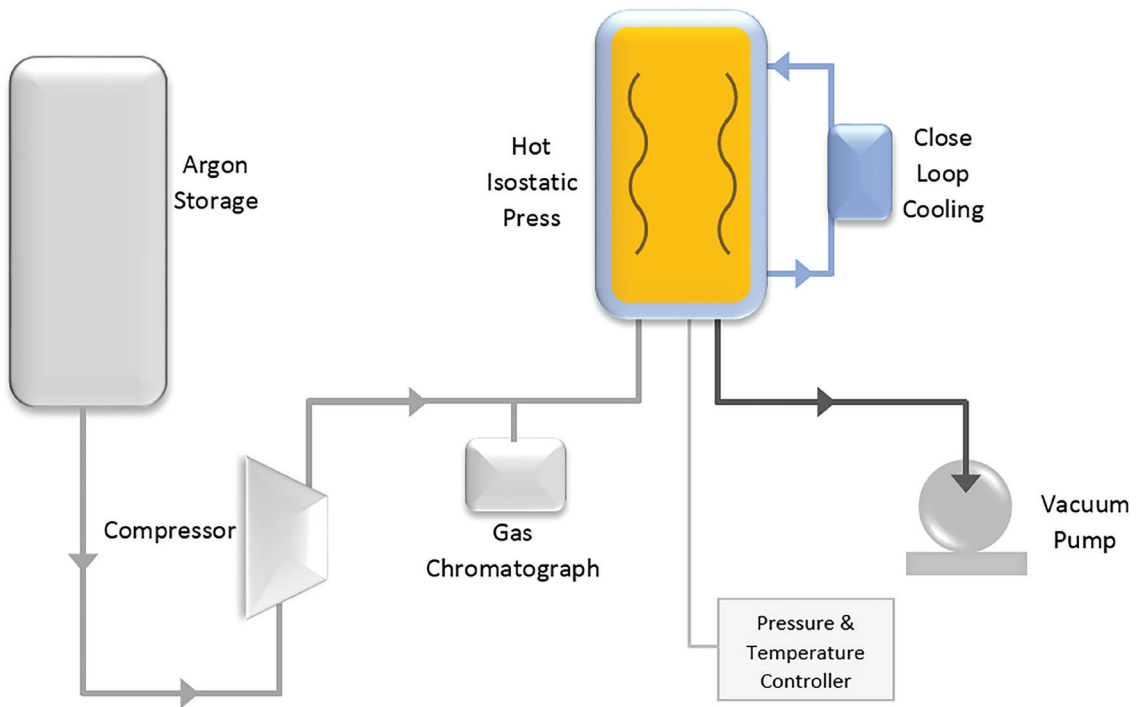
HIP installations will process many tons of titanium, aluminium, steel and super-alloy castings, removing porosity and improving the performance of parts such as turbine blades and oilfield components. In almost all cases metals in any state require heat treatment to improve their properties, if components 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 H<sub>2</sub>-O<sub>2</sub>-N<sub>2</sub>-CH<sub>4</sub>-CO-CO<sub>2</sub>-NMHC-H<sub>2</sub>O in UHP argon.

### **HIP typical installation:**



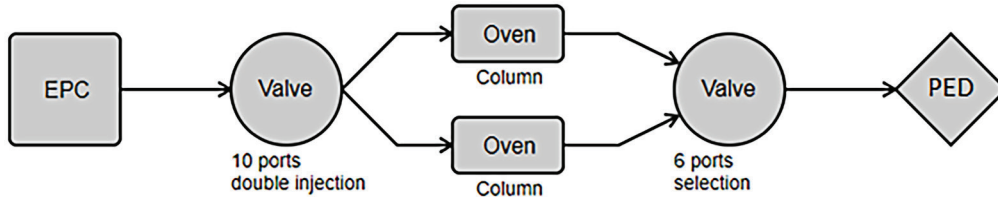
### **LDETEK SOLUTION:**

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.

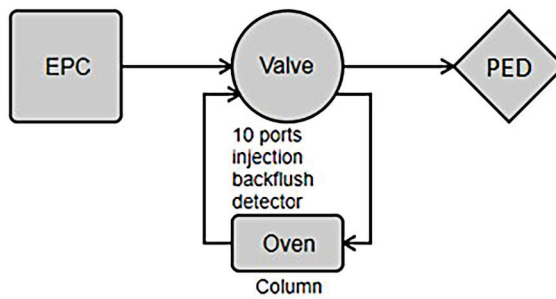
**Block 1 is used to measure trace H2-O2-N2-CH4-CO and CO2**

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**

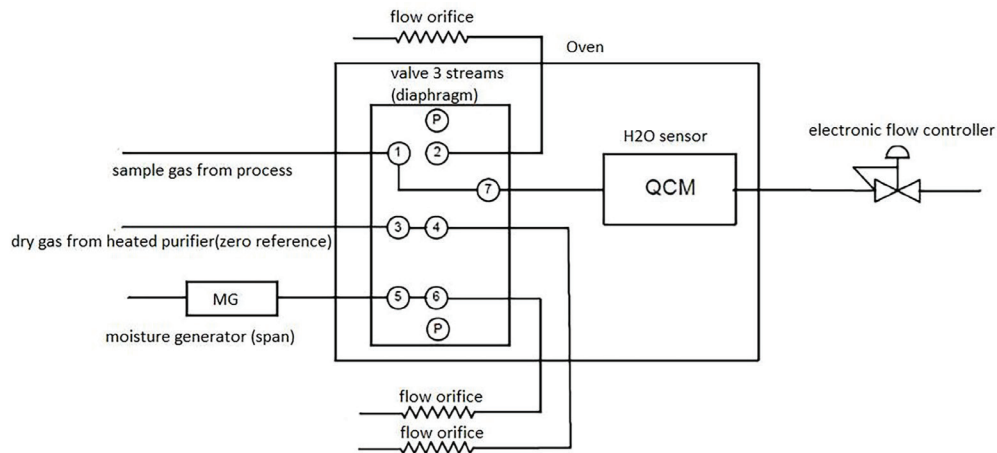
The block 2 arrangement allows to inject light impurities to vent and by reverting the valve position, the C2s-C3s-C4s hydrocarbons are grouped together as one peak to form NMHC to the PED. The PED has the right optic, selective to hydrocarbons.



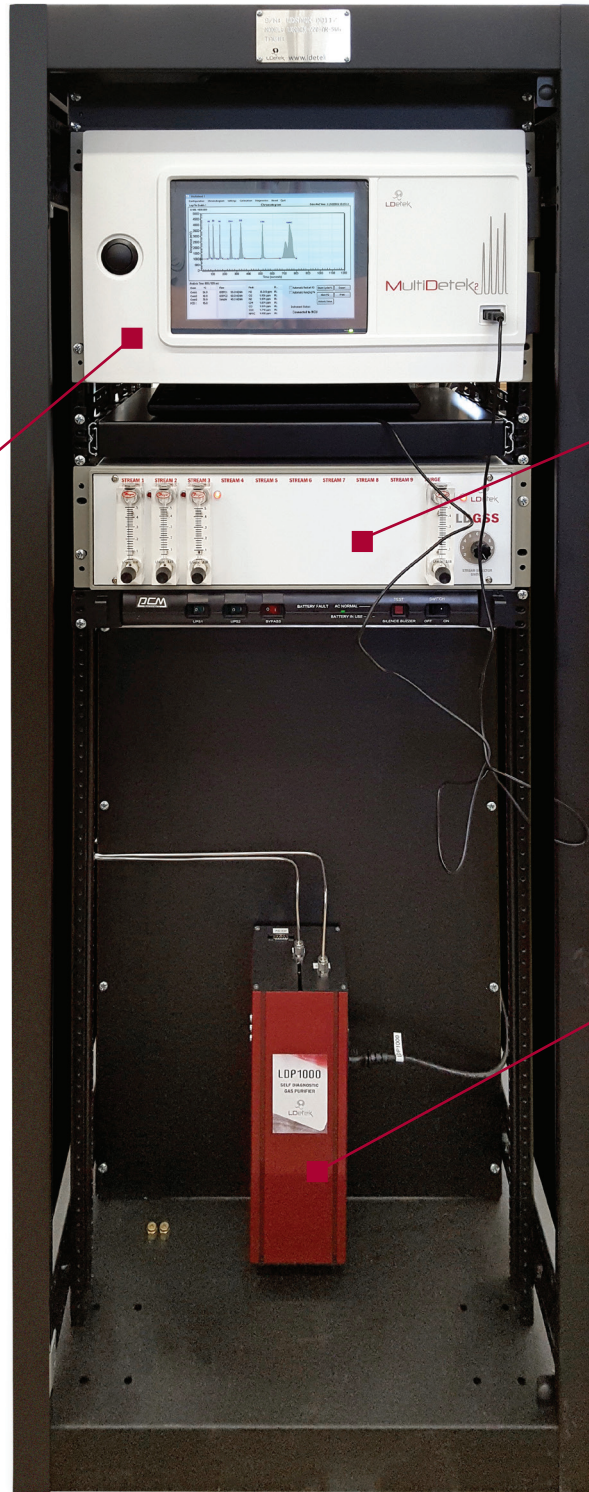
For the analysis of trace H2O, 2 solutions are offered depending of the requirements.

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 Idl 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 H<sub>2</sub>-O<sub>2</sub>-N<sub>2</sub>-CH<sub>4</sub>-CO-CO<sub>2</sub>-H<sub>2</sub>O in UHP argon.

## LDGSS

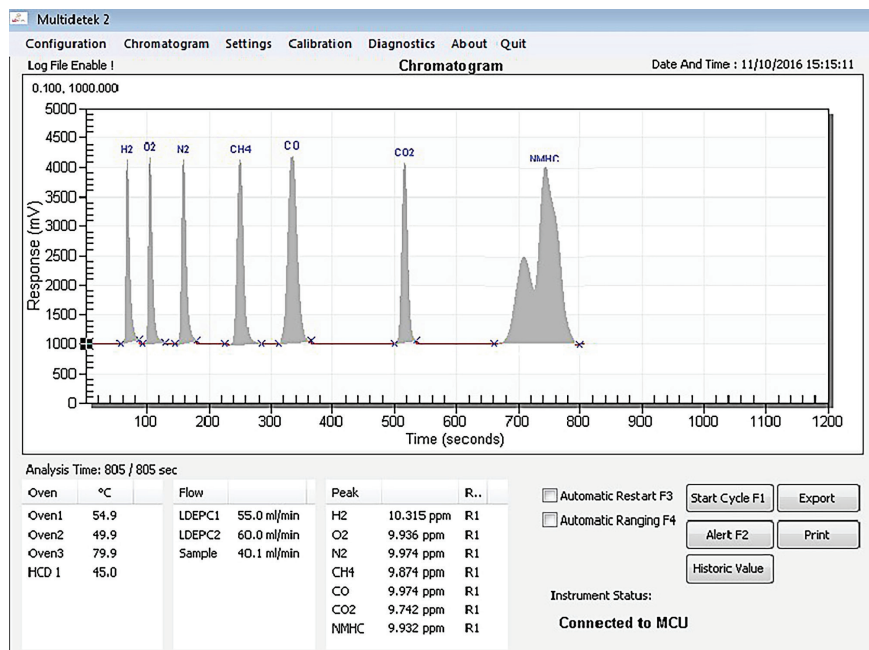
ultra high purity gas stream selector system for switching between the different streams and the span calibration gas (Up to 10 streams in one device is available).

## 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 H<sub>2</sub>-O<sub>2</sub>-N<sub>2</sub>-CH<sub>4</sub>-CO-CO<sub>2</sub> 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 H<sub>2</sub>O makes a reliable turnkey solution for the HIP furnace manufacturers, 3D printer manufacturers and some controlled atmosphere manufacturers.

