

APPLICATION NOTE

LD12-1

Greenhouse analysis with the PlasmaDetek

The popularity to measure greenhouse gases (CH_4 , CO_2 and N_2O) has increased considerably in the last years with the global warming concerns. Chromatography is the well known technique to measure them and different detectors are used to achieve this task. This application note will explain how we can effectively do it with a simple gas chromatograph configuration involving the PlasmaDetek detector.

> PLASMADETEK CONFIGURATION

The PlasmaDetek has the advantage that it can be configured to be more sensitive on some compounds than the others. This selectivity configuration helps the chromatography to be more effective and easier to setup.

For this application, the detector system is configured to be selective on all three impurities with two dedicated outputs signal:

- **Output 1 : N_2O**
- **Output 2 : CH_4 and CO_2**



Figure 1:
PlasmaDetek detector

> CHROMATOGRAPHY CONFIGURATION

Typical configuration to make such measurement requires methanizer, FID and ECD detectors, H_2 Fuel, make up gas and air supply.

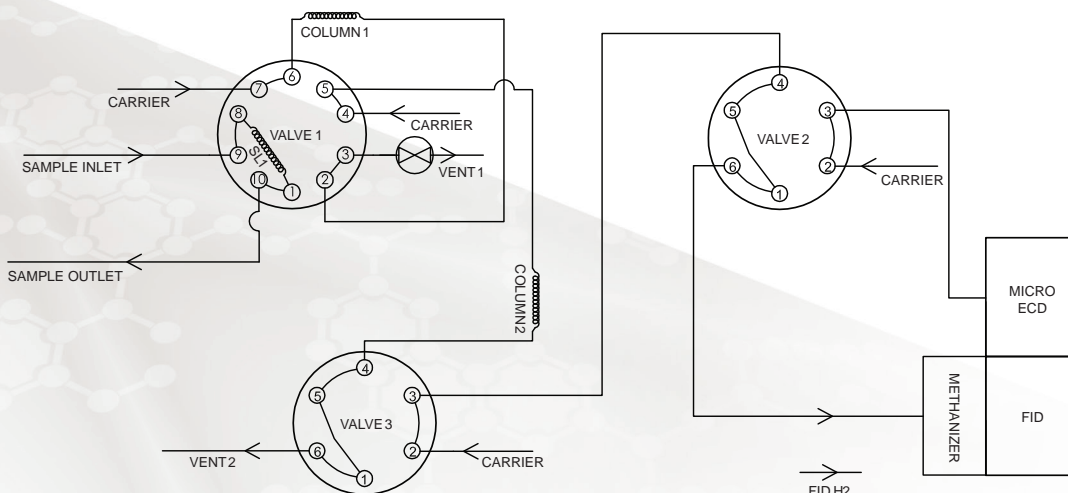


Figure 2:
Typical configuration
for greenhouse measurement

With the PlasmaDetek, only one detector can be used to measure CH₄, CO₂ and N₂O in air. All three components elute in the same detector. That reduces cost and complexity of the system.

Another advantage is the use of argon or helium as carrier gas. Both carriers are suitable and give the performance desired.

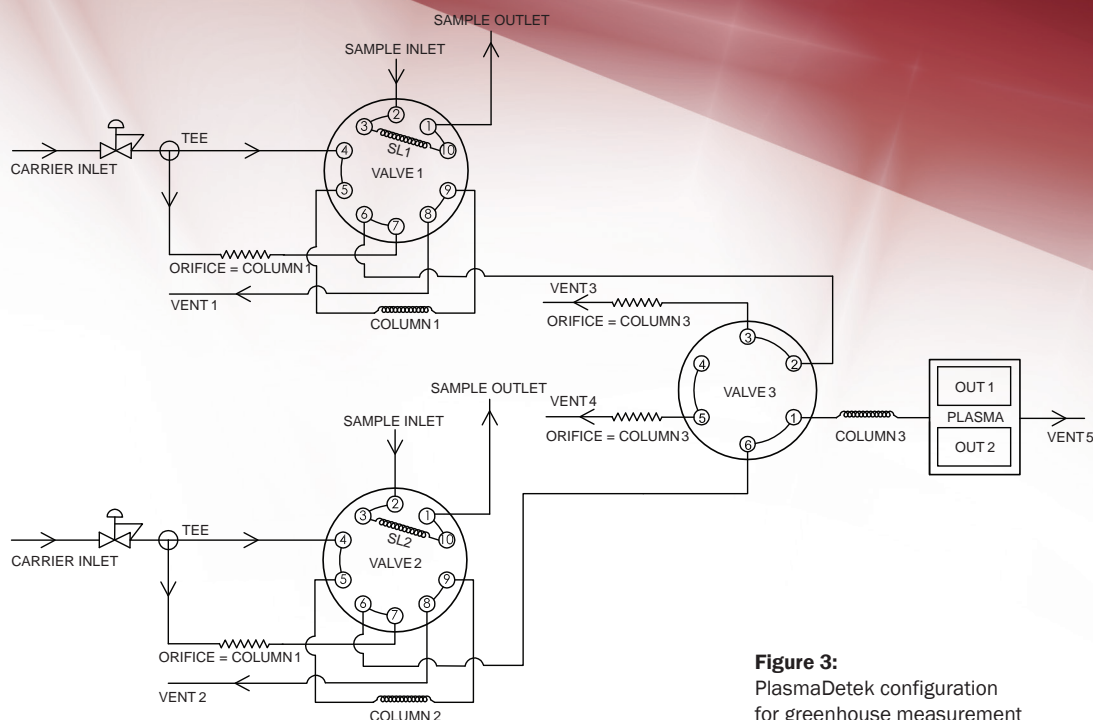


Figure 3:
PlasmaDetek configuration
for greenhouse measurement

Valve 1 is used for CH₄ and CO₂ measurement. The backflush to vent configuration vents out water from the sample with a HayeSep D 100/120 10' (column 1). Valve 3 is used to vent out air before going to the other HayeSep D 100/120 10' (column 3). This second column separates CH₄ and CO₂ from the remaining air.

Valve 2 is also configured in a backflush to vent with a HayeSep D 100/120 10' (column 2). A different sampling loop size is used to be able to measure N₂O. Valve 3 is used to vent out air and CO₂. N₂O is then brought to the detector by itself and can be measured in very low concentration.

> RESULTS AND PERFORMANCE

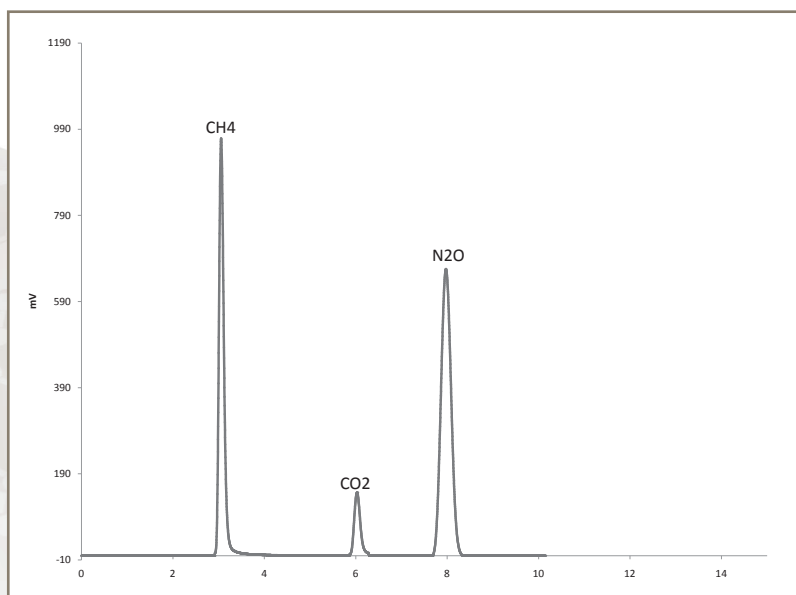


Figure 4: 8 ppm CH₄, 50 ppm CO₂ and 10 ppm N₂O in helium

Figure 4 shows the chromatogram obtained with the LDetek configuration described above, with a 8 ppm CH₄, 50 ppm CO₂ and 10 ppm N₂O standard. This result was used to calculate the LOQ and LOD of each compounds shown in **figure 5**.

Component	Concentration (ppm)	Peak Height	Noise	S/N	LOD (ppb) S/N=3	LOQ (ppb) S/N=5
CH ₄	8	965	0,04	24125	1	1,65
CO ₂	50	143	0,04	3575	42	70
N ₂ O	10	671	0,04	16775	1,8	3

Figure 5: LOQ and LOD calculation

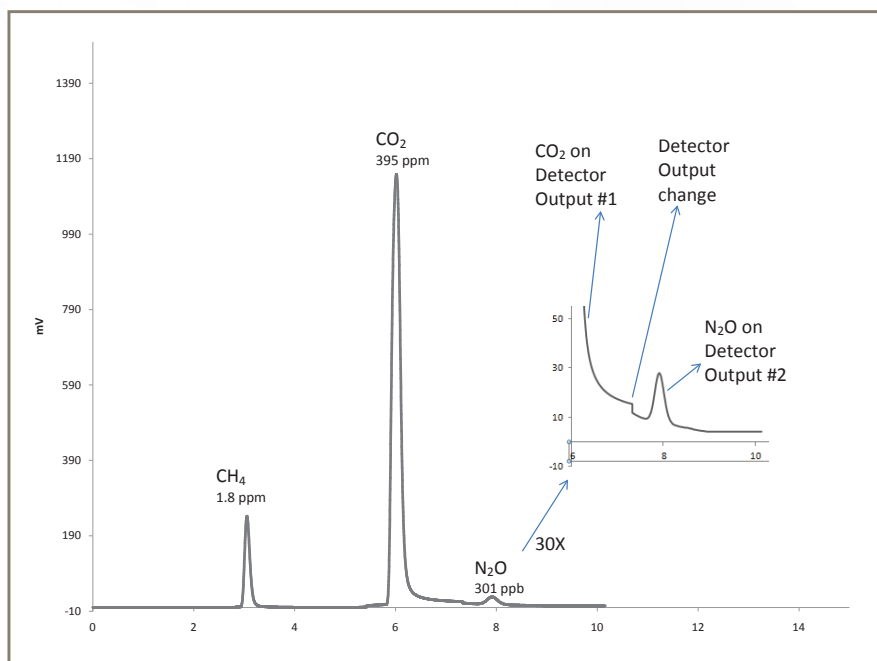


Figure 6: CH₄, CO₂ and N₂O in ambient air

All three components are measured without interference in air providing accurate and very sensitive detection. The LOD of N₂O ensures that its measurement in air is detected easily.

Those results and performances depend on the chromatographic system and conditions of operation.

> CONCLUSION

This technique is simple and cost effective compared to the most common configurations that can be found on the market. No make-up gas, fuel, air, FID and ECD radioactive detector are required to make this measurement. Only one PlasmaDetek detector with two outputs using argon or helium as carrier gas can be used to achieve level of sensitivity needed. The ease of installation and startup of the PlasmaDetek makes it perfectly suitable for this environmental application.



LDetek

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APPLICATION NOTE

LD12-2

Analysis of permanent gases and light hydrocarbons with the PlasmaDetek

The PlasmaDetek is ideal to measure permanent gases and light hydrocarbons in different matrices. Only one detector system is needed to accomplish this task. Such measurement is required in many different applications field: industrial, petrochemical, energy, environmental, etc. The sensitivity, the stability, the ease of start-up and installation make this system very attractive for any users.

> PLASMADETEK CONFIGURATION

The PlasmaDetek is configured with two outputs signal to be able to detect all components. Both argon or helium carrier gas can be used.

- **Output 1 : H₂, C₁ to C₄**
- **Output 2 : O₂, N₂, CO, CO₂**

No need of fuel, air, doping gas, methanizer or other devices with the system. This is a stand-alone detector system that requires only carrier gas to make the measurement of each compound.

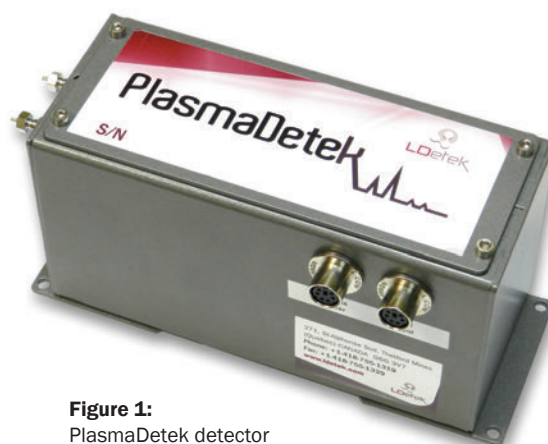
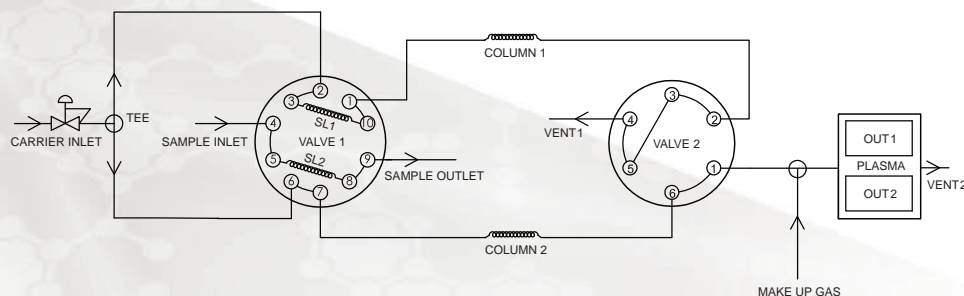


Figure 1:
PlasmaDetek detector

> CHROMATOGRAPHY CONFIGURATION

To make the measurement of all components described above, figure 2 describes an easy configuration. Only one 10 ports injection valve and one selection valve are used. One RT Molecular Sieve 5A 30m x 0.53mm(column 2) separates H₂, O₂, N₂, CH₄, CO before being measured by the detector. The other column, a RT Alumina Bond 30m x 0.53mm x 10mm(column 1), does the same for the CO₂ and C₁ to C₄.



- 1 x 10 ports injection valve
- 1 x 6 ports valve for channel selection
- 1 x RT Alumina Bond 30m x 0.53mm x 10mm (column 1)
- 1 x RT Molecular Sieve 5A 30m x 0.53mm (column 2)
- Argon or helium carrier
- Carrier Flow : 4 cc/min
- Make-up : 60 cc/min
- Detector : PlasmaDetek two outputs

Figure 2:
Plumbing configuration for H₂, O₂, N₂, CO, C₁ to C₄ in Helium or Argon

> RESULTS AND PERFORMANCE

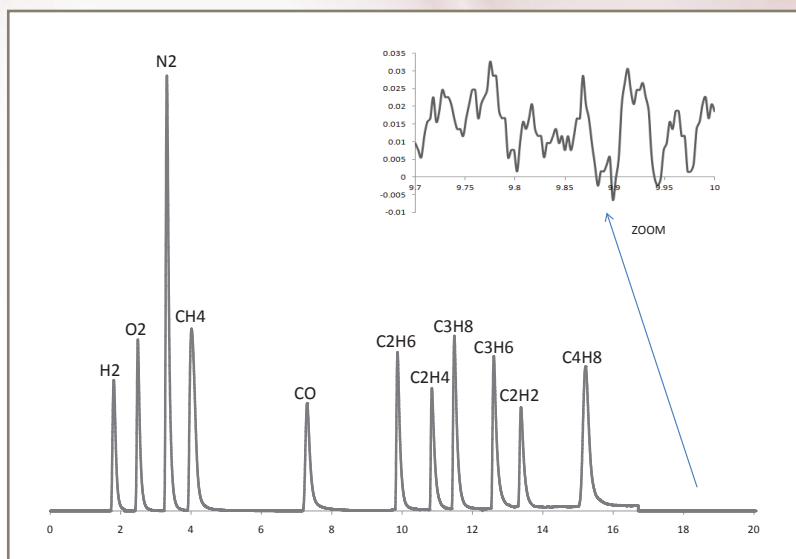


Figure 3: 10ppm H2 - O2 - N2 - CH4 - CO - C2H6 - C2H4 - C3H8 - C3H6 - C2H2 - C4H8

Component	Concentration	Peak Height	Noise	S/N	LOD (ppb) S/N=3	LOQ (ppb) S/N=5
H2	10	342	0,039	8769	3,4	5,7
O2	10	450	0,039	11538	2,6	4,3
N2	10	1142	0,039	29282	1,0	1,7
CH4	10	576	0,039	14769	2,5	4,1
CO	10	282	0,039	7230	4,1	6,9
C2H6	10	402	0,039	10307	2,9	4,9
C2H4	10	330	0,039	8461	3,6	5,9
C3H8	10	442	0,039	11333	2,6	4,4
C3H6	10	426	0,039	10923	2,7	4,6
C2H2	10	274	0,039	7025	4,3	7,1
C4H8	10	366	0,039	9384	6,4	5,4

Figure 4: LOQ and LOD calculation

Figure 3 shows the chromatogram obtained from this configuration. The chromatograph uses output 1 to measure H2 and the HC's and output 2 for the others.

Figure 4 shows the performance of the PlasmaDetek for such components obtained with the configuration described above.

Those results and performances depend on the chromatographic system and conditions of operation.

> CONCLUSION

With the PlasmaDetek, we can measure permanent gases and C1-C4 with only one detector. This is a very cost effective solution in terms of gas supply, plumbing configuration and time consuming. The performance is indisputable. Application in ppb is now easy to make with such configuration.

Selectivity on some impurities can also be configured in the PlasmaDetek to make Chromatography easier when working on different background or focus on some impurities. Heavier hydrocarbons can also be measured. Please contact LDetek for more information.

APPLICATION NOTE

LD12-3

Analysis of argon in pure oxygen with the PlasmaDetek and ArgoTek

The complexity of measuring argon as impurity in chromatography comes from the fact that typical columns on the market do not separate argon and oxygen. Both elute at the same time making the analysis difficult in low concentration. There are typical techniques for this measurement:

- Using an oxygen trap which involves regeneration procedure with H₂ supply, maintenance, consumable and complex chromatography system.
- Cryogenic configuration where columns need to be used in cold environment which involves complex manipulation.
- Using an online oxygen analyzer in parallel and subtract the oxygen from the measurement of Ar+O₂. But this requires a second analyzer and it is difficult to get an accurate measurement in low concentration.

But the combination of the PlasmaDetek and the ArgoTek column is the ideal solution to measure argon in ppt to %.

> PLASMADETEK CONFIGURATION

The PlasmaDetek is configured with one output signal to be selective to argon. The detector system becomes more sensitive to argon than oxygen by at least a factor of 10.

This is a stand-alone detector system that requires only helium carrier gas to make the measurement. No need of doping gas or other devices to make it selective to argon against oxygen.



Figure 1:
PlasmaDetek detector

> CHROMATOGRAPHY CONFIGURATION

This measurement is done by using a simple backflush to vent configuration. The ArgoTek (column 1) is used to separate the argon from oxygen. The argon comes out first of the column and it is sent to the detector. When the argon is out of the ArgoTek, we simply flush outside the oxygen.

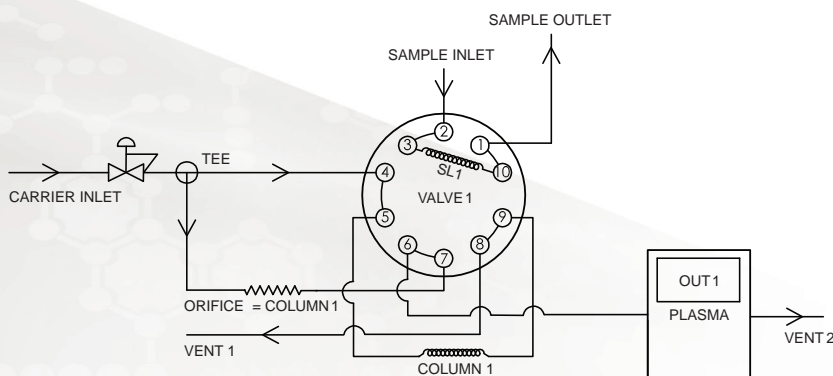


Figure 2:
Plumbing configuration
for argon in pure oxygen

- One PlasmaDetek selective to Argon
- Helium carrier gas: 30 cc/min
- 1 x 10 ports diaphragm valve
- 1 x Argotek column
- 1 x sampling loop: 20 µl



> RESULTS AND PERFORMANCE

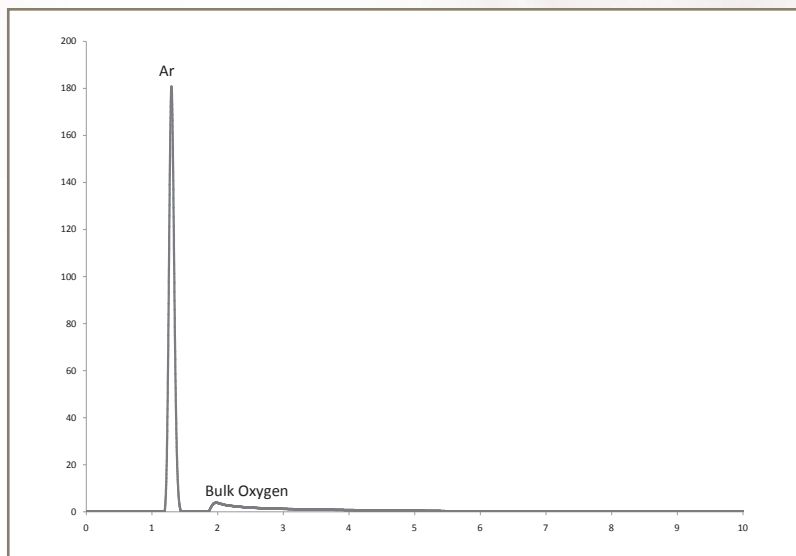


Figure 3: 1 ppm Argon in pure Oxygen

Figure 3 shows the chromatogram obtained from this configuration. The low concentration of argon impurity is easily separated from the oxygen bulk. But more importantly, the selectivity of argon with the detector, gives a better separation of the two compounds, which allows a faster recovery time.

Component	Concentration	Peak Height	Noise	S/N	LOD (ppb) S/N=3	LOQ (ppb) S/N=5
Ar	1	180	0,039	4615	0,65	1

Figure 4: LOQ and LOD calculation

Figure 4 shows the performance of the system obtained with the configuration described above. This PlasmaDetek and ArgoTek combination gives ppt limit of detection with no oxygen interference. Higher concentration, up to %, can be also measured by simply changing the sample volume.

> CONCLUSION

Using the Argotek and PlasmaDetek combination, it becomes very easy to measure this compound compared to other available technologies. This is a cost effective, maintenance free configuration and quick analysis technique avoiding consumables and regeneration procedures that will reduce complexity of the chromatography system.

APPLICATION NOTE

LD12-4

Analysis of nitrogen in hydrogen and oxygen bulk with the PlasmaDetek

Measuring nitrogen as impurity in low concentration is not an easy task. It is mostly the case in hydrogen and oxygen background. Even if the chromatography system is quite efficient, the remaining bulk gas could influence the reading of nitrogen.

The PlasmaDetek, configured to be selective on nitrogen, brings very good result for this measurement. This document will demonstrate how this technology can help to make better analysis on nitrogen with any gas chromatograph system.

> PLASMADETEK CONFIGURATION

The PlasmaDetek has the advantage that it can be configured to be more sensitive on some compounds than the others. This selectivity configuration helps the chromatography to be more effective and easier to setup.

By using proper optical system, the detector becomes selective on nitrogen. No need of doping gas or other devices to make it selective. There is no other such detector on the market that will give this nitrogen selectivity.

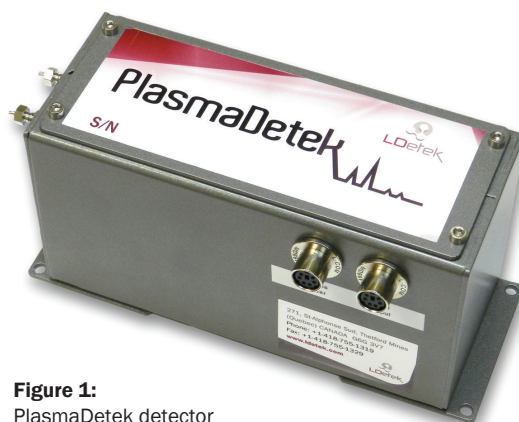


Figure 1:
PlasmaDetek detector

> CHROMATOGRAPHY CONFIGURATION

A simple backflush configuration is used to make the measurement of N_2 in H_2 or O_2 . Column 1 will initiate a separation of H_2 or O_2 from N_2 and bulk gas will be vented out as much as possible. Before N_2 goes out, the flow is directed to column 2. Both carrier gases can be used: argon or helium.

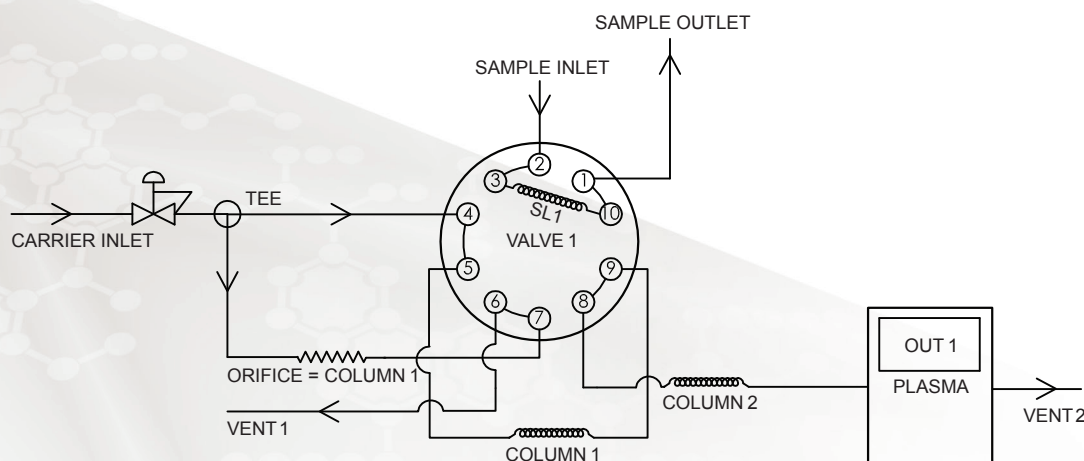


Figure 2:
Backflush configuration
with two columns

> RESULTS AND PERFORMANCE

Using a 5 ppm standard nitrogen in bulk hydrogen as sample, two chromatograms were generated with the chromatography configuration described above with a helium carrier. **Figure 3** is using a non-selective detector and **figure 4** the PlasmaDetek N₂ selective configuration. As it is clearly shown, the selectivity from the PlasmaDetek is improving the chromatogram. Almost no hydrogen is seen by the detector.

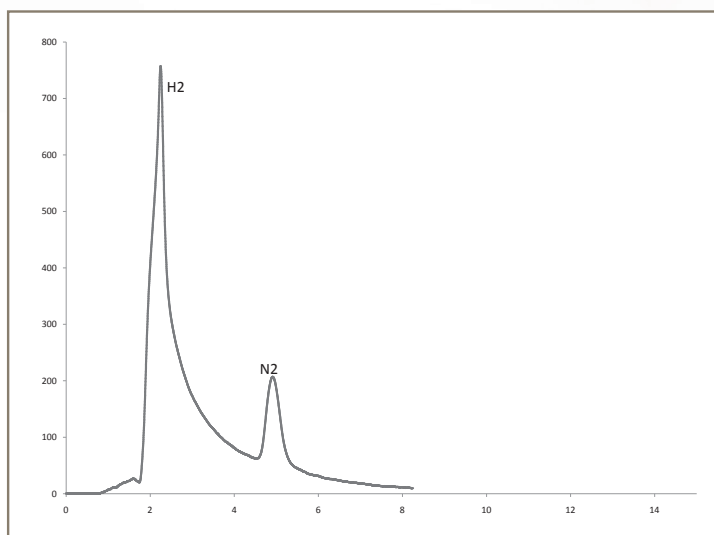


Figure 3: 5 ppm N₂ in H₂ with non-selective detector system

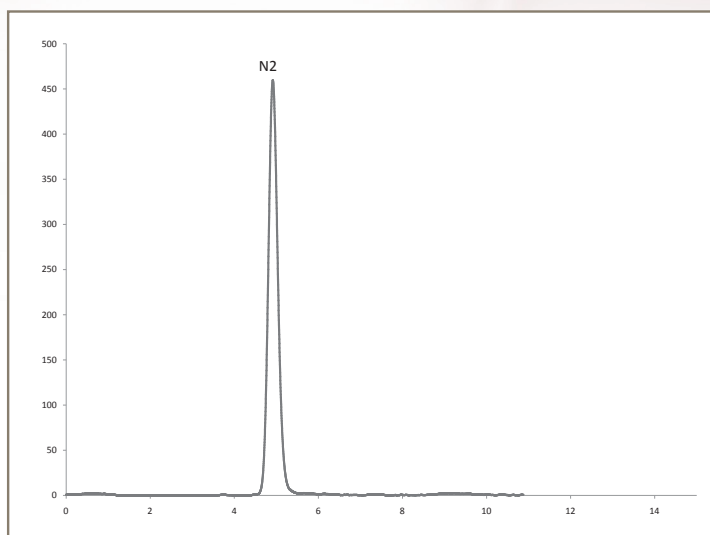


Figure 4: 5 ppm N₂ in H₂ with PlasmaDetek selective system

Same procedure was done with a standard of 420 ppb nitrogen in oxygen in similar backflush configuration with helium carrier. **Figure 5** and **figure 6** show the comparison with a non-selective detector and the PlasmaDetek N₂ selective configuration. It is also obvious in this case that selectivity helps to have better results and less drifting problem for the peak from remaining bulk.

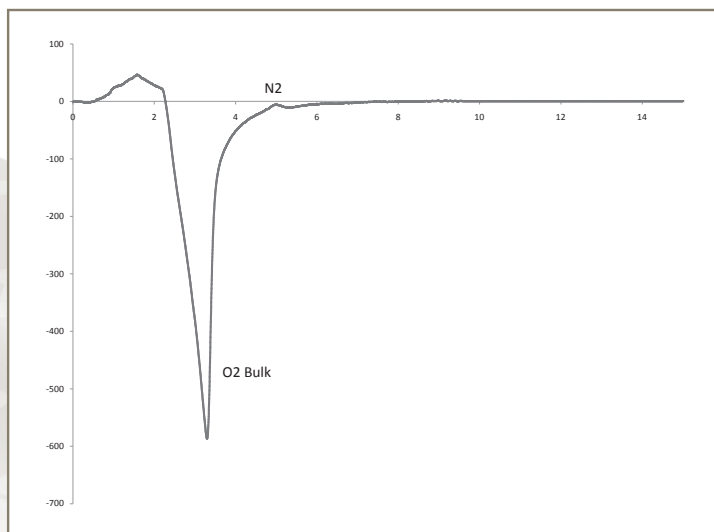


Figure 5: 420 ppb N₂ in O₂ with non-selective detector system

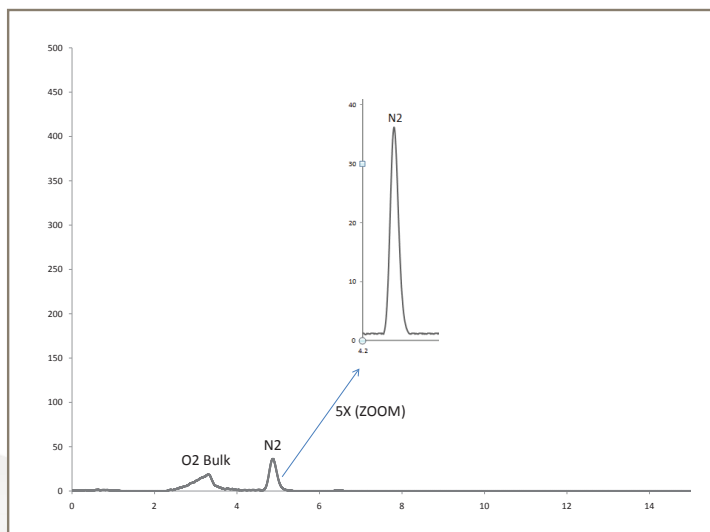


Figure 6: 420 ppb N₂ in O₂ with PlasmaDetek N₂ selective system

Having 79% of nitrogen in air, It is important to ensure that the chromatographic system is leak free. Low concentration nitrogen requires good hardware components to avoid any inboard and outboard leakages. It is even more the case, when measuring in pure H₂ or O₂.



Figure 7: High performance diaphragm valve

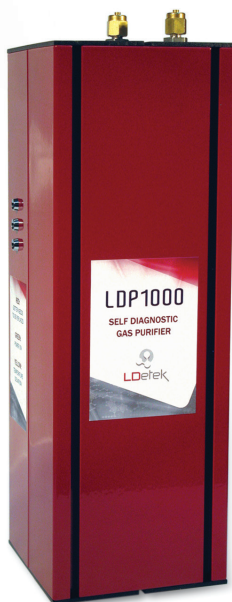


Figure 8: LDP1000 noble gas purifier

Good quality carrier gas is also mandatory to get analysis in low concentration. With its unique specific design, the LDP1000 noble gas purifier is a cost effective device for any gas chromatograph. It eliminates any contamination problems coming from the gas source.

In **figure 9**, we have evaluated the performance of both analysis. With the fact that the PlasmaDetek has a very low noise and a good response on the nitrogen, low concentration can be measured. LOD and LOQ can vary depending on sample volume or condition of operation. < 1 ppb LOD can also be obtained by optimizing the system.

Component	Concentration (ppm)	Peak Height	Noise	S/N	LOD (ppb) S/N =3	LOQ (ppb) S/N=5
N ₂ in H ₂	5	455	0,038	11973	1,25	2,1
N ₂ in O ₂	0,42	38	0,038	1000	1,26	2,1

Figure 9: LOQ and LOD calculation

> CONCLUSION

This is the ideal configuration to measure nitrogen in different matrices. By having such selectivity, you can reduce analysis time and make fast chromatography. In some cases, consumables such as traps can be avoided. It becomes a cost effective solution, maintenance free system and can give better limit of detection by reducing residual background effect.

The PlasmaDetek can also be configured with multiple selective outputs to allow measurements of different compounds. Please contact LDeTek for more information.



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APPLICATION NOTE

LD12-06



Increasing argon production with the MultiDetek



▲ MultiDetek2

▲ PlasmaDetek2

Air is composed of nitrogen (78.09%), oxygen (20.94%) and argon (0.934%). To produce pure argon, distillation process separates the air constituent by the use of distillation columns. Such installation on an air separation plant is used for many years.

This separation process is based on vapor pressure of each component. Argon is taken from a low pressure column and introduced in a second column called crude argon. Since argon vapor pressure is close to oxygen, and also between nitrogen and oxygen, its extraction is between those two other components in the low pressure column before being introduced in the second column.

However, the level of nitrogen can be problematic when introducing the extracted gas to the crude argon column. Introducing high volume of nitrogen in the crude argon column will make it stop working. Dumping phenomenon can also arise from the crude argon column.

The extraction of argon in the low pressure column is critical, the goal is to get as much as possible argon and reduce the nitrogen level. Figure 1 shows the vapor pressure of each compound in such column. The maximum level of argon has too much nitrogen. A compromise between argon and nitrogen level must be done.

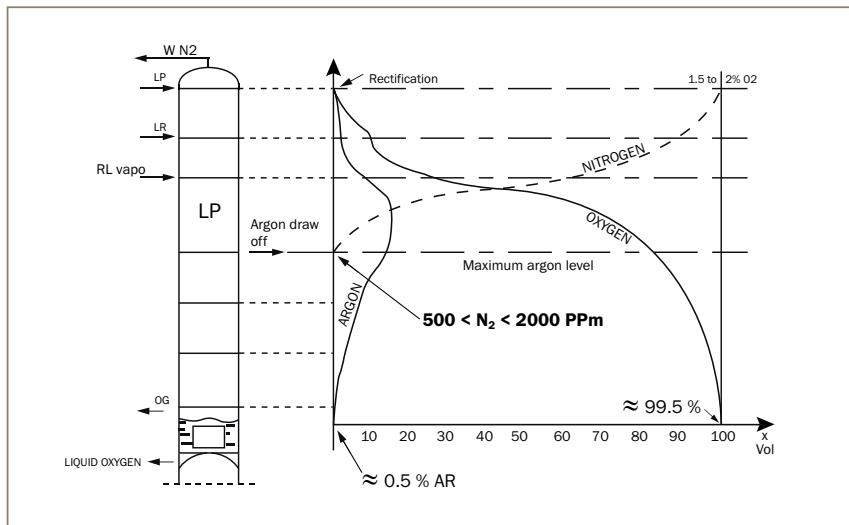


Figure 1

Most of the plants will then take argon at a point where it is safe to keep nitrogen level low. Nitrogen is lower, but argon as well. Oxygen is then relatively high in the crude argon column.

Typical analytical tools are the oxygen and argon measurement. Nitrogen can be approximated and you keep the introduction of nitrogen at minimal level.

Such analysis method gives a safe system, but not optimal argon production can be achieved.

WHAT CAN BE DONE ?

By using proper analytical tool to measure nitrogen level in the low pressure column, the plant can reach the maximum level of argon extraction with the acceptable nitrogen in the crude argon column. The LDetek's MultiDetek is the ideal tool to measure nitrogen in oxygen/argon mixture. By measuring continuously nitrogen in such mixture, optimal argon production can be achieved.

Signifiant increase of argon production can be achieved just by measuring nitrogen level accurately.

The MultiDetek can be interfaced with any acquisition system on ASU. Analog signal or digital signal can be provided. Ethernet connection is also available.

Such analyzer is easy to operate with its user friendly interface and can operate 24/24 to ensure extraction is well tuned up.

Such MultiDetek is using the PlasmaDetek technology with nitrogen selectivity. This avoids any oxygen trap consumable.

CONCLUSION:

The pay back of such analytical tool is fast. It will operate for many years and gives optimal efficiency of any argon production plant. The combination of MultiDetek platform with the PlasmaDetek technology is a reliable, efficient and accurate system that any plant is looking for.



APPLICATION NOTE

LD12-7

Analysis of Neon-Hydrogen-Argon-Krypton-Nitrogen with the PlasmaDetek & the HSR-Etek column

The analysis of Neon, Hydrogen, Argon, Krypton and Nitrogen by chromatography has always been problematic. The bad separation and the poor sensitivity for the analysis of these impurities are the reasons that make it complicated to realize. Even with the use of capillary columns, cryogenic system and/or hydrogen trapping system, the analysis of low ppb of these impurities cannot be performed in one run. Furthermore, the detectors available in the industry have some sensitivity limitation. It is then very difficult to measure low ppb for the mentioned impurities especially for Neon with the existing technologies.

LDetek has then developed a method for the analysis of Neon, Hydrogen, Argon, Krypton and Nitrogen. Using the PlasmaDetek system and the HSR-Etek column, the low ppb analysis without any interference can be successfully realized using a simple injection.

> PLASMADETEK SYSTEM

The PlasmaDetek can be configured in a non selective or in a selective mode depending of the lowest detection limit requested and the interference gases. Using its selective mode, the response ratio between the analyte of interest and the interference gases will be increased. The detector can then be very sensitive to the Neon, Hydrogen, Argon, Krypton and Nitrogen without being affected by background gases or interference gases.

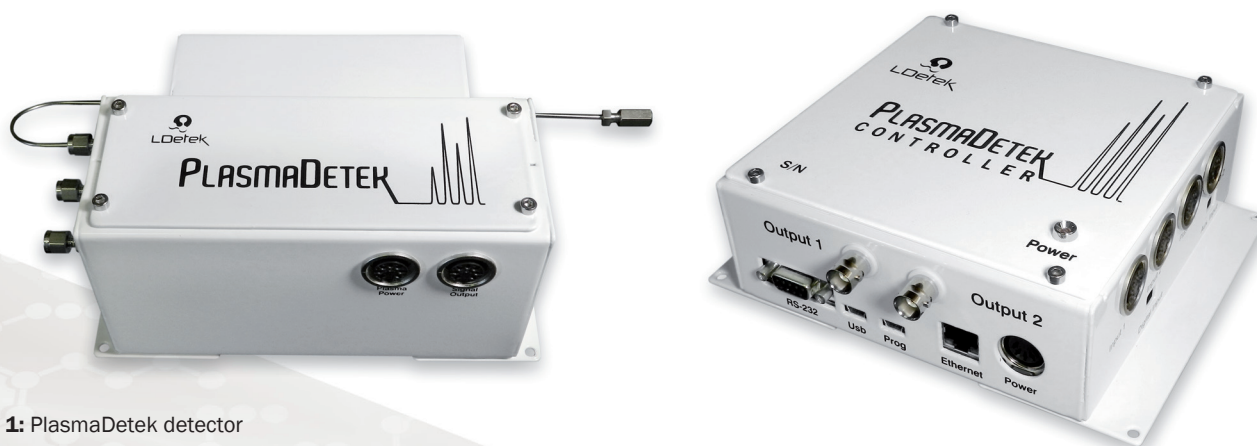


Figure 1: PlasmaDetek detector

This is a stand-alone detector system that requires only helium or argon carrier gas to make the measurement. No need of doping gas or other devices. By using argon as carrier gas, the analysis of neon and argon cannot be realized.

> CHROMATOGRAPHY CONFIGURATION

This measurement is done by using a simple injection (see figure 2)). The sample is injected through the HSR-Etek column and then goes directly to the PlasmaDetek inlet port. The carrier flow rate must be regulated at 30sccm and maintained stable. The column temperature must operate at 45Celsius until minute 4. The column temperature is then ramped up to 90Celsius at 10Celsius/minute. Using the temperature ramping mode, the analysis of Krypton and Nitrogen can be included in less than 16 minutes.

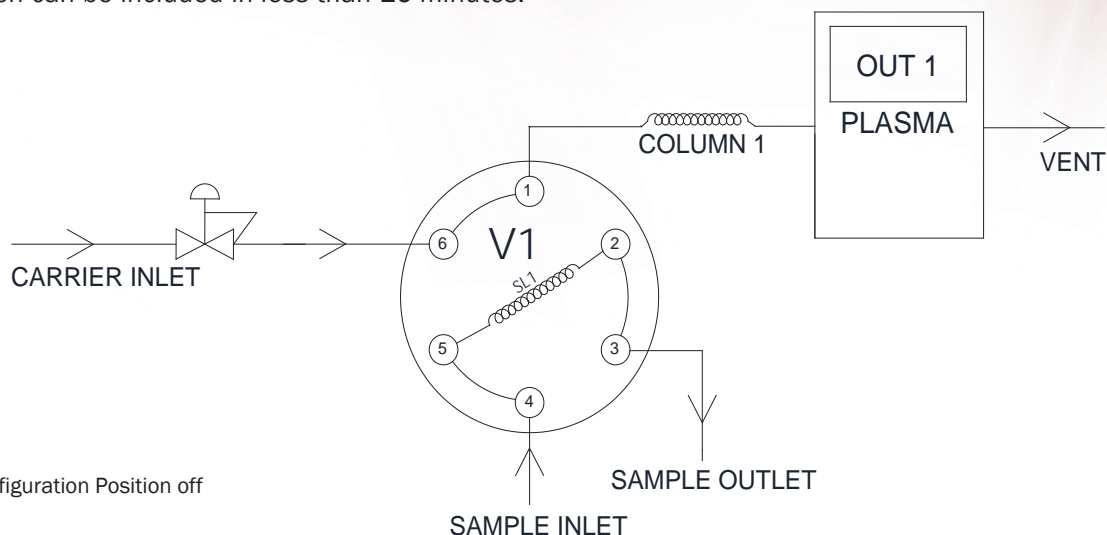


Figure 2: Configuration Position off

> RESULTS AND PERFORMANCE

Figure 3 shows the chromatogram obtained from this configuration.

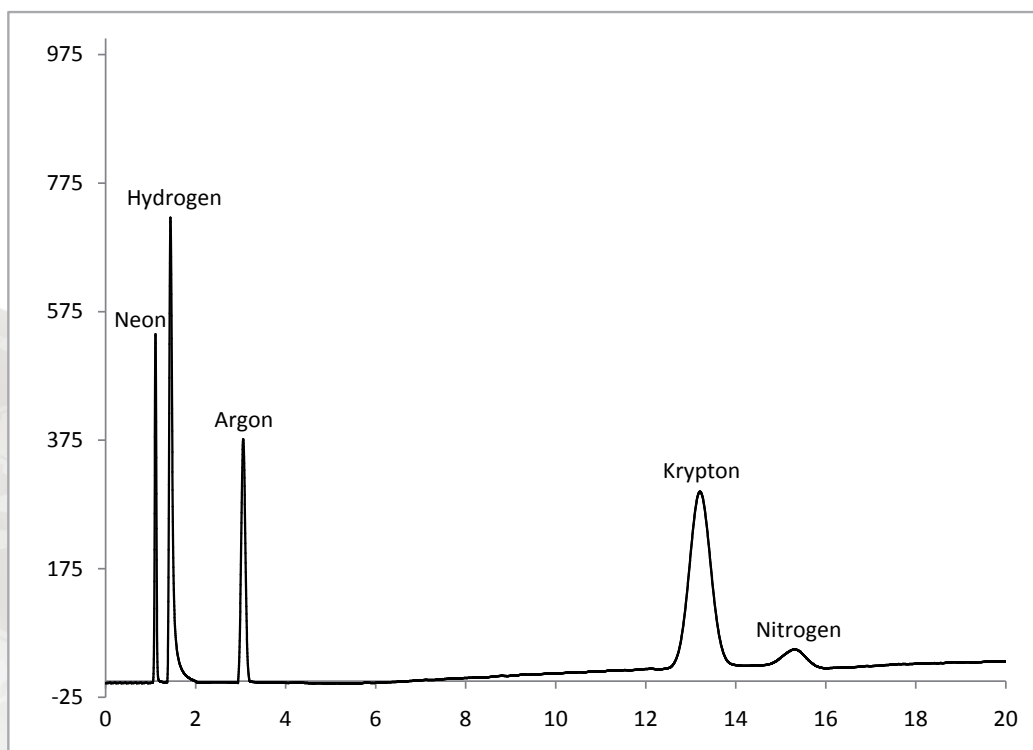


Figure 3:
Mixture 4ppm Neon,
5ppm Hydrogen,
1ppm Argon,
4ppm Krypton,
0.5ppm Nitrogen
in balance Helium

Figure 4 shows the performance of the system obtained with the configuration described above. The PlasmaDetek and HSR-Etek combination gives the possibility to achieve low ppb limit of detection with good resolution for Neon/Hydrogen, Argon/Oxygen and Krypton/Nitrogen using a simple injection. Higher concentration, up to %, can be also measured with the same system.

Component	Concentration (ppm)	Peak Height	Noise	S/N	LOD (ppb) S/N =3	LOQ (ppb) S/N=5
Neon	4	562	0.049	11469	1.04	1.74
Hydrogen	5	756	0.049	15428	0.97	1.62
Argon	1	381	0.049	7775	0.39	0.64
Krypton	4	341	0.049	6959	1.72	2.87
Nitrogen	0.5	38	0.022	1727	0.86	1.45

Figure 4: LOQ and LOD calculation

> PLASMADETEK SELECTIVITY

Using its selectivity, the PlasmaDetek gives the advantage of being very sensitive to desired impurities only. See below an example of analysis of 2ppm Krypton and 4ppm Nitrogen in a balance of Oxygen using HSR-Etek column.

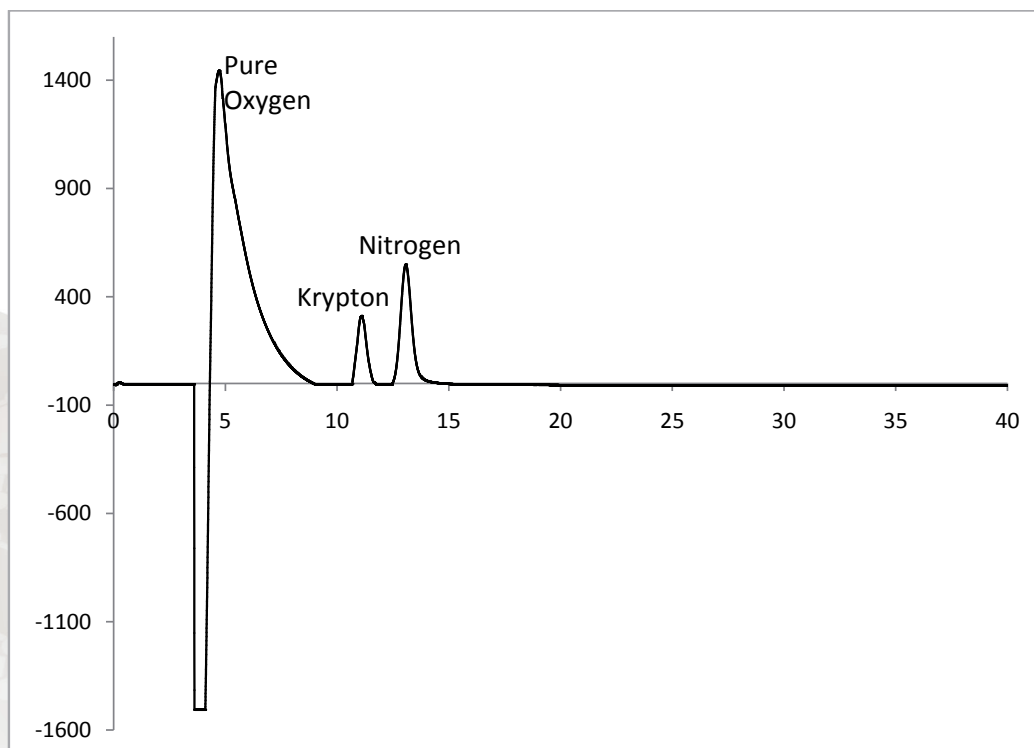


Figure 5:
Mixture 2ppm Krypton,
4ppm Nitrogen
in balance Oxygen

> CONCLUSION

Using the combination of the PlasmaDetek and the HSR-Etek column, the analysis of Neon, Hydrogen, Argon, Krypton and Nitrogen becomes an easy task. A simple injection through the HSR-Etek column and to the PlasmaDetek allow to measure ppb, ppm or percentage level of the mentioned compounds. This is a cost effective, maintenance free system and quick analysis technique avoiding consumables and regeneration procedures that will reduce complexity of the chromatography system.



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APPLICATION NOTE

LD12-8

Analysis of Sulfurs with the PlasmaDetek

The analysis of sulfurs can now be performed using the PlasmaDetek technology. With its sulfurs selective mode, the analysis of low ppb sulfurs can be easily quantified.

> PLASMADETEK CONFIGURATION

The PlasmaDetek is configured with one output signal to be selective to sulfurs.

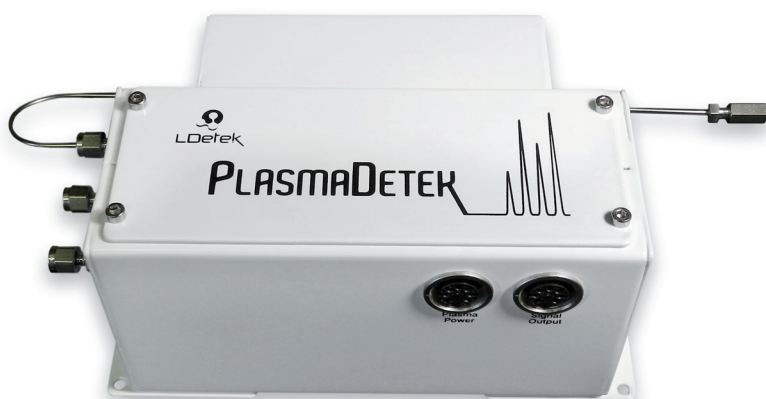


Figure 1: PlasmaDetek detector

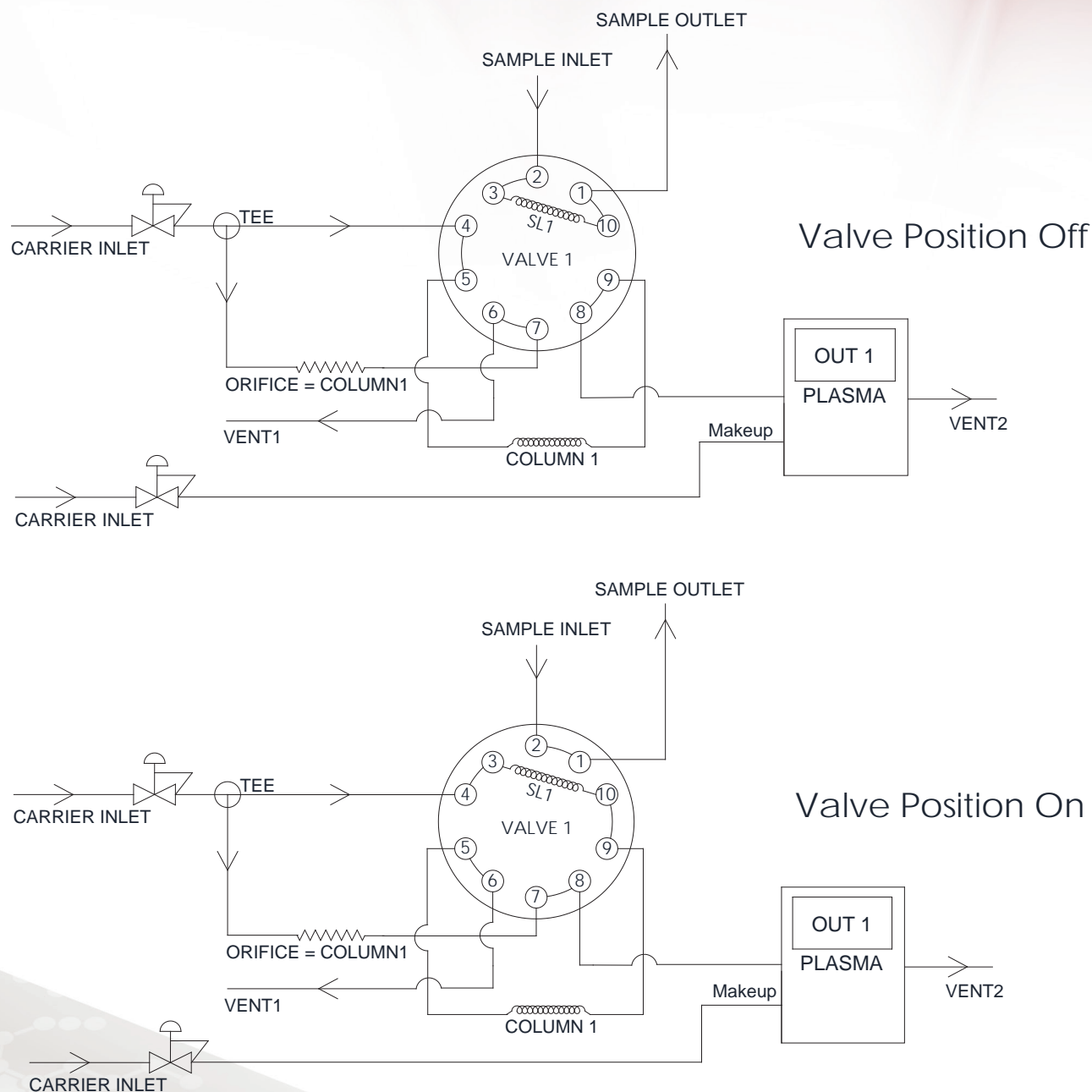


This is a stand-alone detector system that requires only **argon or helium** as carrier gas to make the measurement. No need of doping gas or other devices to make it selective to sulfurs.

> CHROMATOGRAPHY CONFIGURATION

The detector can be used with different configurations to run the sulfurs analysis. As example, the H₂S and COS analysis in balance nitrogen has been tested using a 2 meter RT-XLSulfur column from Restek with a backflush to detector configuration using a 10 ports diaphragm valve. The carrier flow rate has been adjusted to 5sccm using helium (**Argon can also be used as carrier gas to reduce operational cost**).

The diaphragm valve is first set to "position on" to allow the sample injection through the chromatographic column. Most of the nitrogen and/or air are then vented out of the system. The valve position is hold to "position on" until the major parts of the nitrogen and/or air have been vented out. The valve is then set to "position off" just before the hydrogen sulfide elution time to avoid venting it out. The sulfurs impurities then flow back through the 2 meter RT-XLSulfur and goes one by one to the PlasmaDetek for analysis.



- One PlasmaDetek selective to sulfurs
- Helium carrier gas: 5 cc/min
- 1 x 10 ports diaphragm valve
- 1 x 2 meters RT-XLSulfur micro packed column
- 1 x sampling loop: 250 μ l

Figure 2: plumbing configuration for analysis of H₂S & COS in balance Nitrogen

> RESULTS AND PERFORMANCE

Figure 3 shows a chromatogram run with this configuration with a sample containing ppm H₂S and COS in a nitrogen balance. The chromatogram demonstrates the high sensitivity and selectivity to sulfurs in comparison with pure nitrogen.

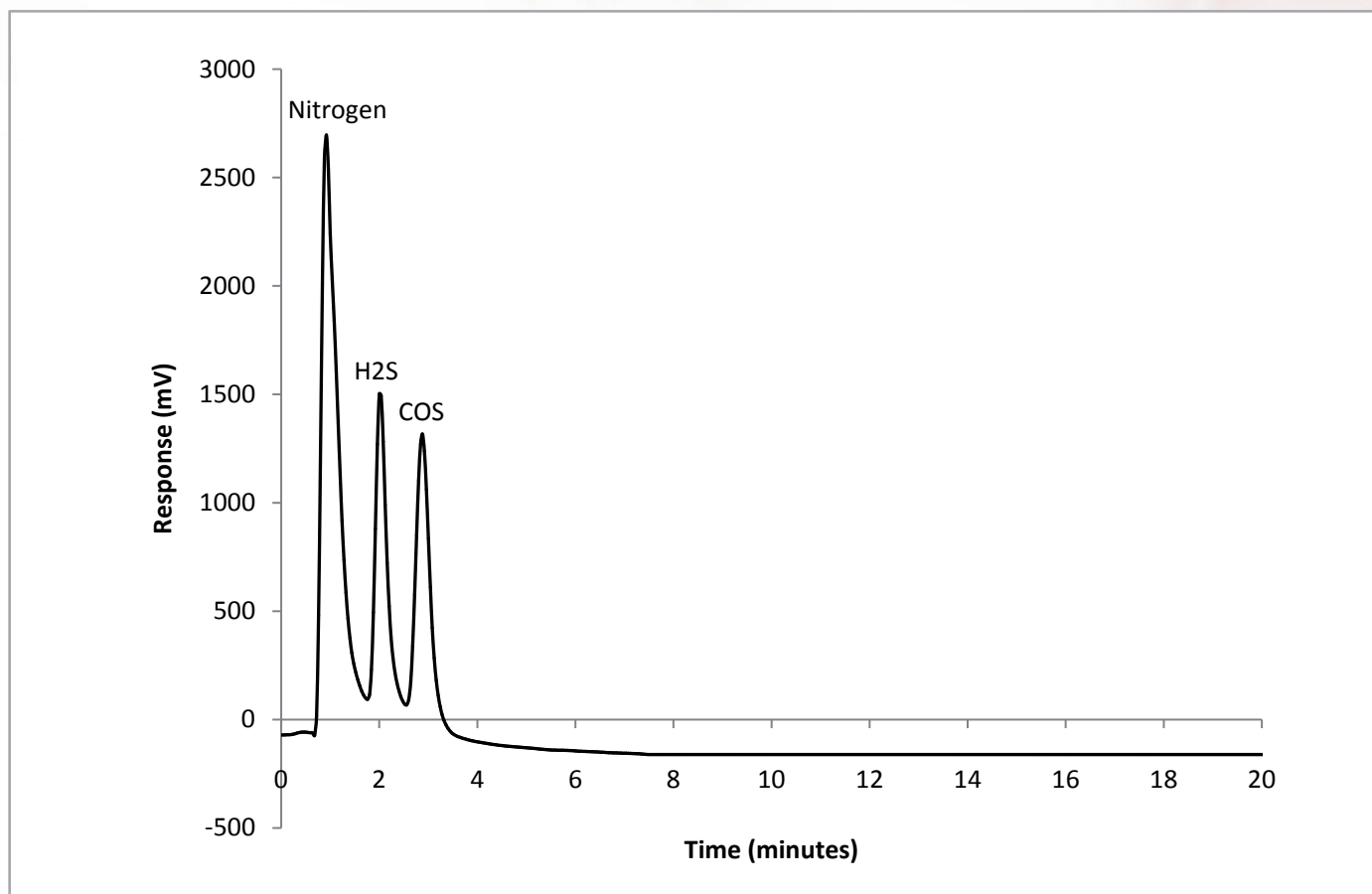


Figure 3: Mixture 10ppm H₂S, 9ppm COS in balance Nitrogen

Figure 4 shows the performance of the PlasmaDetek system obtained with the configuration described above. The results demonstrate the high level of sensitivity to sulfurs.

Component	Concentration (ppm)	Peak Height	Noise	S/N	LOD (ppb) S/N=3	LOQ (ppb) S/N=5
Hydrogen sulfide	10	1410	0.054	26111	1.10	1.9
Carbonyl sulfide	9	1302	0.054	24111	1.11	1.8

Figure 4: LOQ and LOD calculation

> APPLICATIONS

The PlasmaDetek can be easily installed in any laboratory, industrial or portable gas chromatography system. For the sulfurs analysis, the use of a portable unit is often required since the GC system needs to be moved at different locations. The ability of the PlasmaDetek to offer a quick purging and stabilization makes it ideal for portable unit like demonstrated on figure 5.

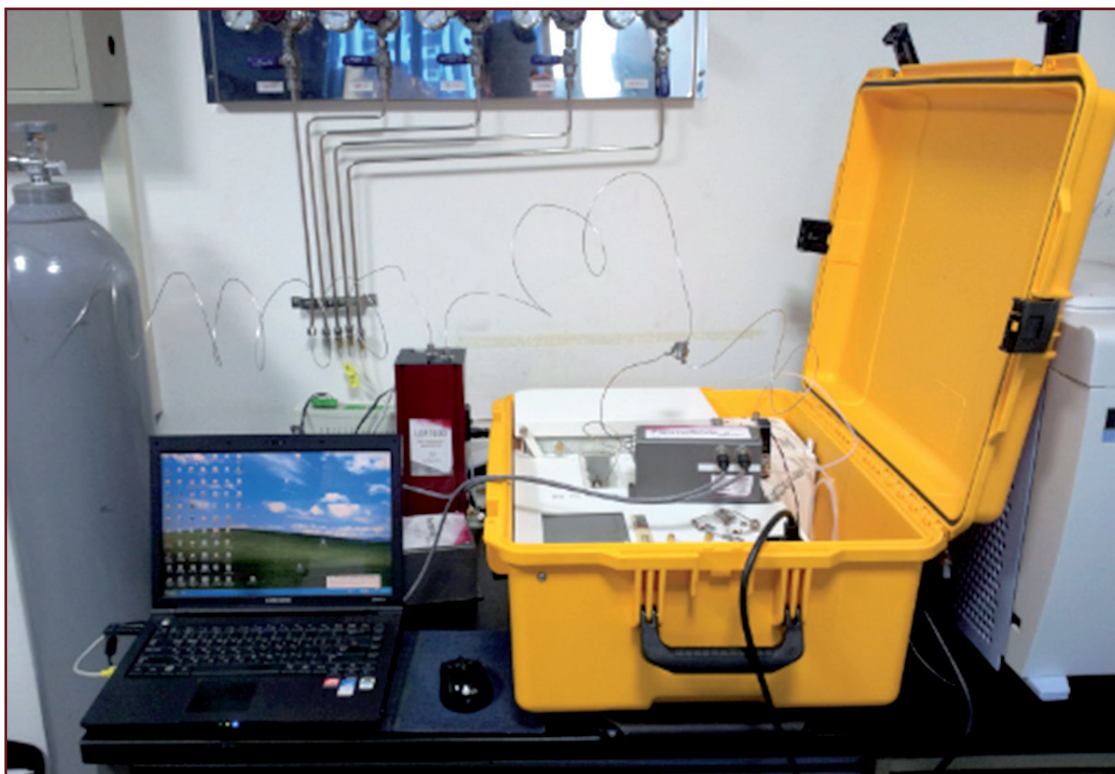


Figure 5: PlasmaDetek installed in a portable unit

> CONCLUSION

Using the PlasmaDetek, the analysis of sulfurs can be performed with success. The possibility to use argon or helium as carrier gas gives more flexibility and allows reducing operational cost. Its ability to be very sensitive to sulfurs simplifies the chromatographic configuration. This is a cost effective and maintenance free system that offers many benefits.



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APPLICATION NOTE

LD12-9

Hydrocarbons measurement for Oxygen production using PlasmaDetek & Multidetek-2



▲ Multidetek-2

◀ PlasmaDetek detector

> INTRODUCTION

Oxygen is one of the basic chemical elements. In its most common form, oxygen is a colorless gas found in air. It is one of the life-sustaining elements on Earth and is needed by all animals. Oxygen is also used in many industrial, commercial, medical, and scientific applications. It is used in blast furnaces to make steel, and is an important component in the production of many synthetic chemicals, including ammonia, alcohols, and various plastics. Oxygen and acetylene are combusted together to provide the very high temperatures needed for welding and metal cutting.

The most common commercial method for producing oxygen is the separation of air using either a cryogenic distillation process or a vacuum swing adsorption process. Nitrogen and argon are also produced by separating them from air. The figure 1 represents a common cryogenic distillation process for producing oxygen.

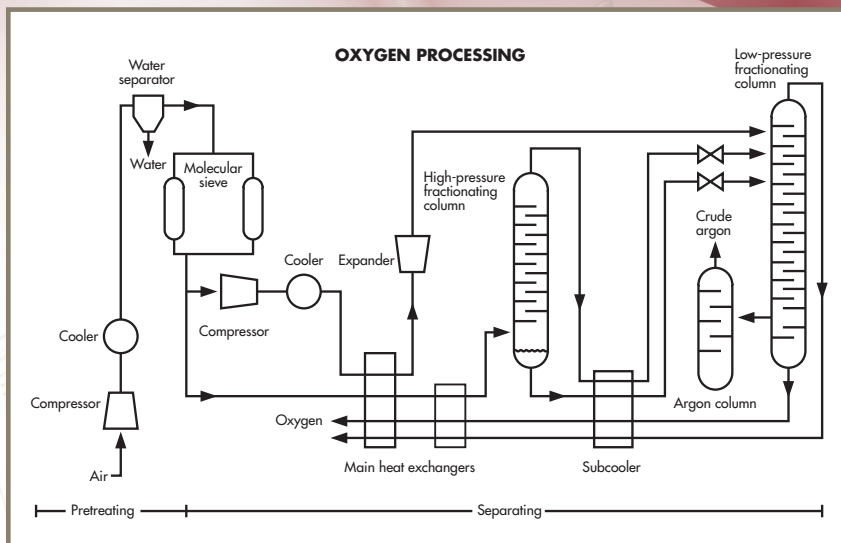


Figure 1:
diagram of a cryogenic distillation process

Because this process utilizes an extremely cold cryogenic section to separate the air, all impurities that might solidify—such as water vapor, carbon dioxide, and certain heavy hydrocarbons—must first be removed to prevent them from freezing and plugging the cryogenic piping what could result to an hazardous situation. The removal of hydrocarbons is also very important to avoid any problem in the subsequent air distillation that could lead to explosion.

Then, the operation of cryogenic distillation air separation units must be monitored by automatic analytical instruments. As a result, their output is consistent in quality and ensures safety of the site. Periodic sampling and analysis of the final product ensures that the standards of purity are being met. A good analytical instrument is then necessary to monitor the various sampling points of the air separation unit.

> THE SOLUTION

With its integrated plasma technology, The LDetek's Multidetek-2 becomes the ideal tool to measure the purity of the oxygen. Its capability to monitor the hydrocarbons level from ppb level up to high ppm level using its plasma detector gives the ideal alternative to the standard use of a FID detector. With the use of the plasma for monitoring the hydrocarbons, there is no more need of using Fuel/Air mixture additionally to the carrier gas. The Multidetek-2 and its plasma only require argon carrier gas. It is then a big advantage since there is no handling and storage of hydrogen anymore on site. Moreover, the use of argon as carrier gas is also a great benefit because the argon is low cost and is already produced and available on any air separation plant.

The use of the Multidetek-2 for monitoring hydrocarbons level in oxygen production is also the ideal tool for a safety point of view. It is critical to have a reliable unit able to monitor 24/7 basis the level of hc's in oxygen on ASU. Being very sensitive to hydrocarbons, including C2H2 which is the most critical component, the Multidetek-2 becomes the best tool to use.

The Multidetek-2 unit can have multiple configurations to allow the measurement of different sampling points of the air separation unit. The monitoring of quality control at the end of oxygen production process can then be easily realized with a configuration allowing low ppb detection. This will ensure an accurate reading necessary for producing high quality grade oxygen. Using the same unit, a higher scale configured for high ppm measurement necessary to monitor the different sampling points of the crude material is also integrated in the unit.

An analysis example of hydrocarbons measurement in pure oxygen appears on the figure 2.

The Multidetek-2 can be interfaced with any acquisition system on ASU. Analog signal or digital signal can be provided. Ethernet connection is also available for remote control. Such analyzer is easy to operate with its user friendly interface and can operate 24/24 to ensure good oxygen production. For this application, such Multidetek-2 is using the PlasmaDetek technology with its hydrocarbons selective detector. This avoids any consumable.

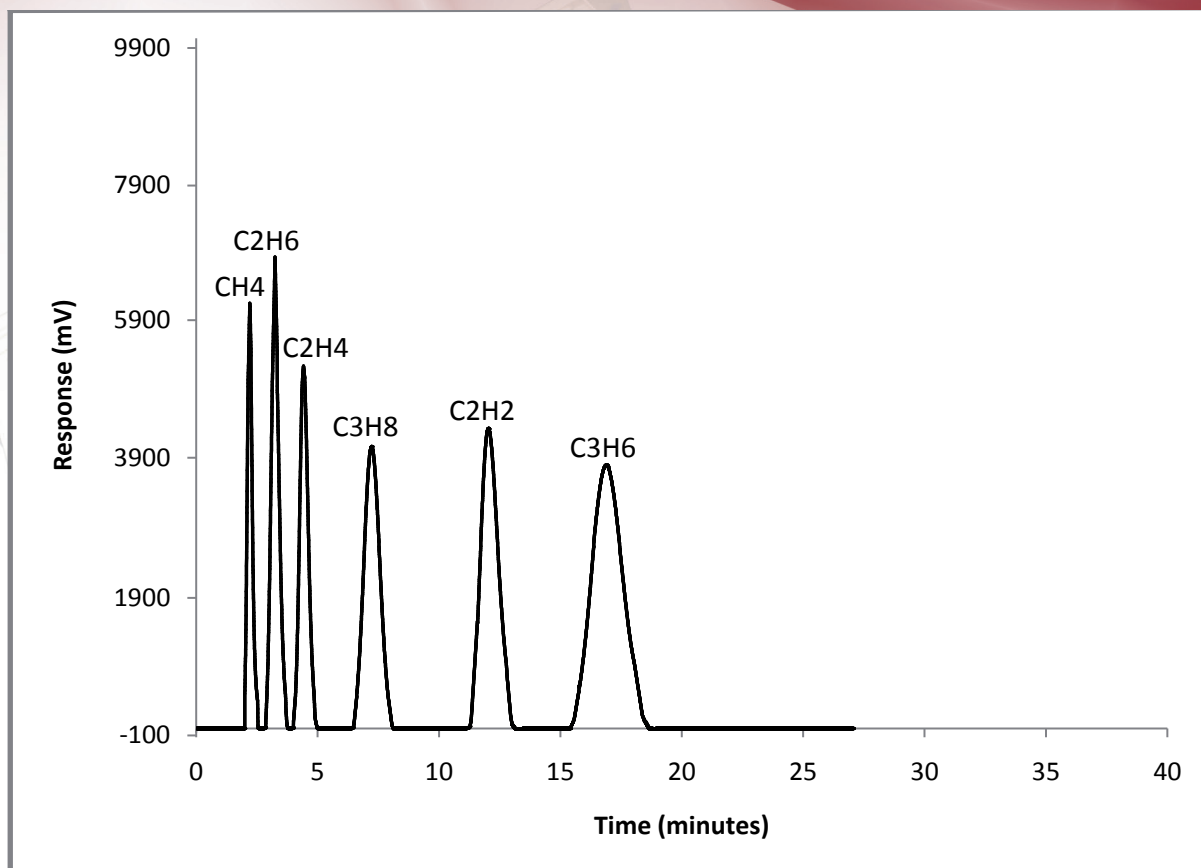


Figure 2: Chromatogram of hydrocarbons in pure oxygen using PlasmaDetek and Multidetek-2

> MORE POSSIBILITIES

A good return on investment can be also realized by configuring the same instrument for more components:

The analysis of CO₂ and any other permanent gases or sulfurs impurities can be added to the same instrument. Even more, the Multidetek-2 also offers the possibility to have multiple background gas configurations. The analysis of argon and nitrogen purity can then be added to the same unit.

One more time, with the simple use of argon as carrier gas and its integrated plasma, the Multidetek-2 can handle multiple measurements in multiple background gases.

> CONCLUSION

The pay back of such analytical tool is fast. It requires only argon as carrier gas which is available at low cost on any air separation plant. There is no more need of handling and storing hydrogen what is usually more expensive due to the need of safety sensors and procedures on site. The PlasmaDetek use in the Multidetek-2 is maintenance free and is a clean detector. It requires no cleaning procedure. The Multidetek-2 will operate for many years and gives optimal efficiency of any oxygen production plant.

The combination of Multidetek-2 platform with the PlasmaDetek technology is a reliable, efficient and accurate system that any plant is looking for.



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APPLICATION NOTE

LD13-01



Analysis of UHP Hydrogen production using Plasmadetek-2 & compact GC Multidetek-2



The high purity hydrogen production demand is rising quickly and the need of measuring low ppb trace in a quick analysis run is then required. Most of the GC technologies available on the market use the same methods for years which required quite complex systems. Those systems require the use of different detectors to cover the application and a complex chromatograph configuration what make the price of such system increasing. The complexity of the chromatograph operations, the long analysis time and the limitations to achieve low ppb measurement are often faced.

THE SOLUTION:

With its integrated plasma technology, The MultiDetek-2 from LDetek becomes the ideal tool to measure the hydrogen purity. Its capability to monitor the permanent gases and hydrocarbons from ppb level up to high ppm level using only one plasma detector gives the ideal solution. In comparison to the existing method, the PlasmaDetek-2 gives the advantage of being selective to the desired impurities and then block the interference coming from the matrix gas. In the case of UHP hydrogen production, measuring low ppb traces of O₂ and N₂ is complex since their elution time is very close to the hydrogen matrix.

Then, to allow the analysis of O₂ and N₂, relatively long molecular sieve columns are required in combination with multiple valves for flushing out the hydrogen gas. In some cases, a hydrogen trapping module may be necessary. It then increases the cost of the system and extends the analysis time. Peaks shape can be also affected with the use of longer columns.

With the PlasmaDetek-2, a simple system having one valve and one Molecular Sieve column is required. The detector is configured with two sensors to measure O_2 and N_2 respectively in selective mode. Refer to Figure 1 for the configuration drawing. Going this way, the analysis time can be reduced, the sensitivity increased and it gives an easy to use system with no consumable or maintenance required.

An analysis example of impurities O_2 & N_2 in a hydrogen sample, using a standard ionization detector in combination with the same configuration that appears on Figure 1, has been used to generate the chromatogram that appears on Figure 2. We can clearly see the interference of the hydrogen over the traces O_2 and N_2 . The hydrogen matrix interference overlapping the traces O_2 - N_2 will be amplified when going lower at low ppb level and it will become not possible to measure the impurities without using another techniques requiring more long columns, additional valves and H_2 trap.

At the opposite, on Figure 3 appears a chromatogram of traces O_2 and N_2 with the use of the PlasmaDetek-2 using its selective mode. It is clearly demonstrated that the selectivity gives an important benefit that makes such type of analysis possible with a simplified chromatography method demonstrated on Figure 1.

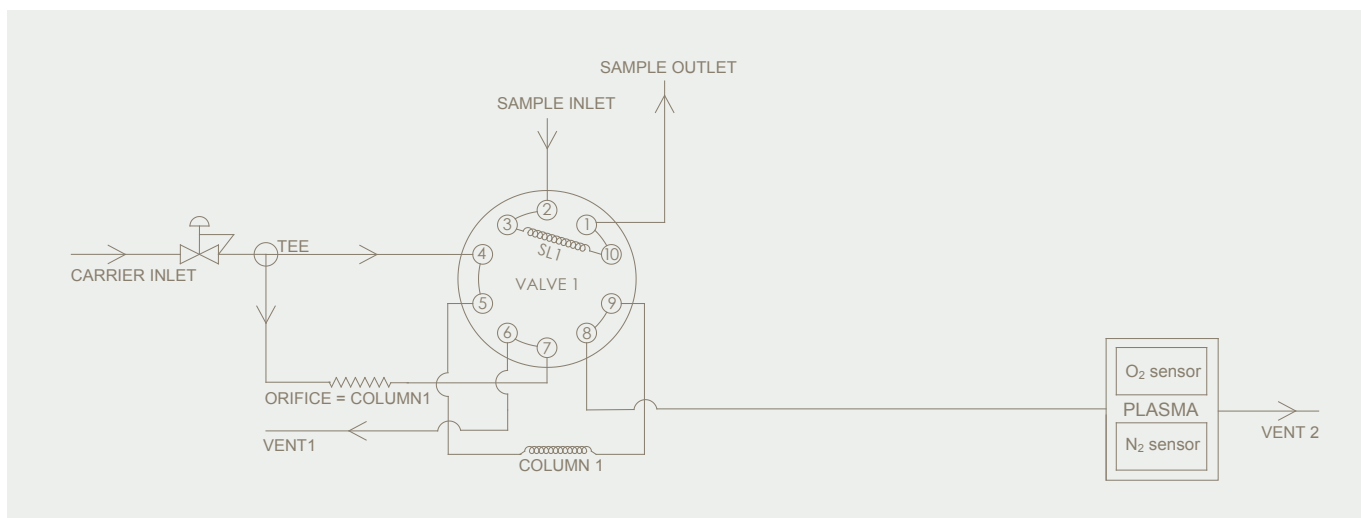


Figure 1 : Configuration used for measuring traces O_2 - N_2 in matrix hydrogen.
(One additional non selective sensor can be added to the same plasma for measuring traces CH_4 - CO with the same channel.)

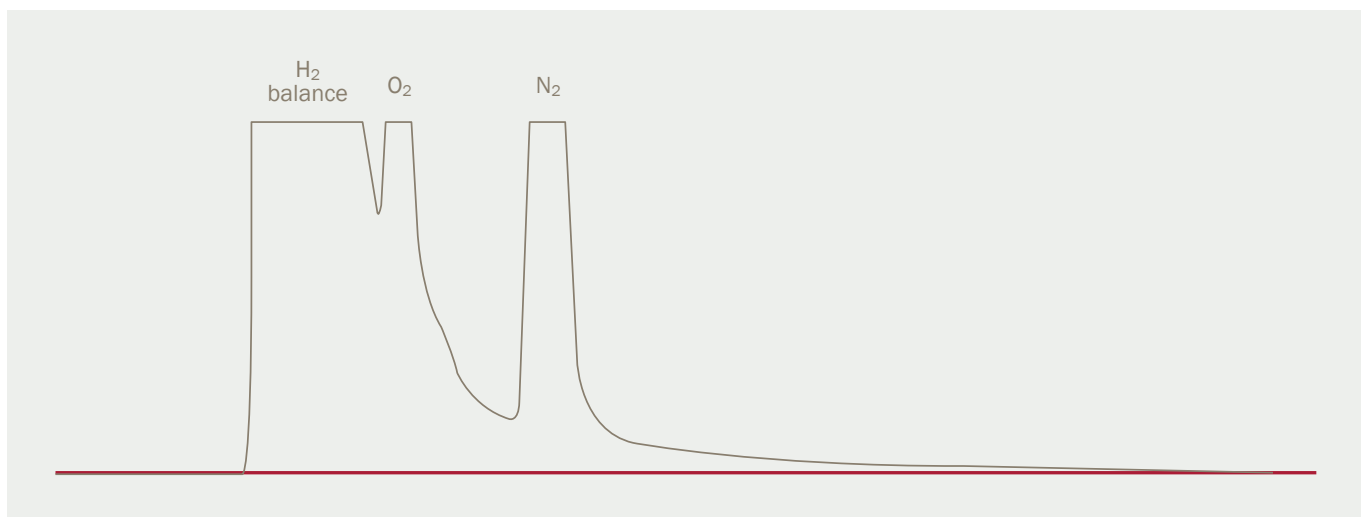


Figure 2 : Chromatogram of traces O_2 - N_2 in a hydrogen matrix using a conventional non selective ionization detector.

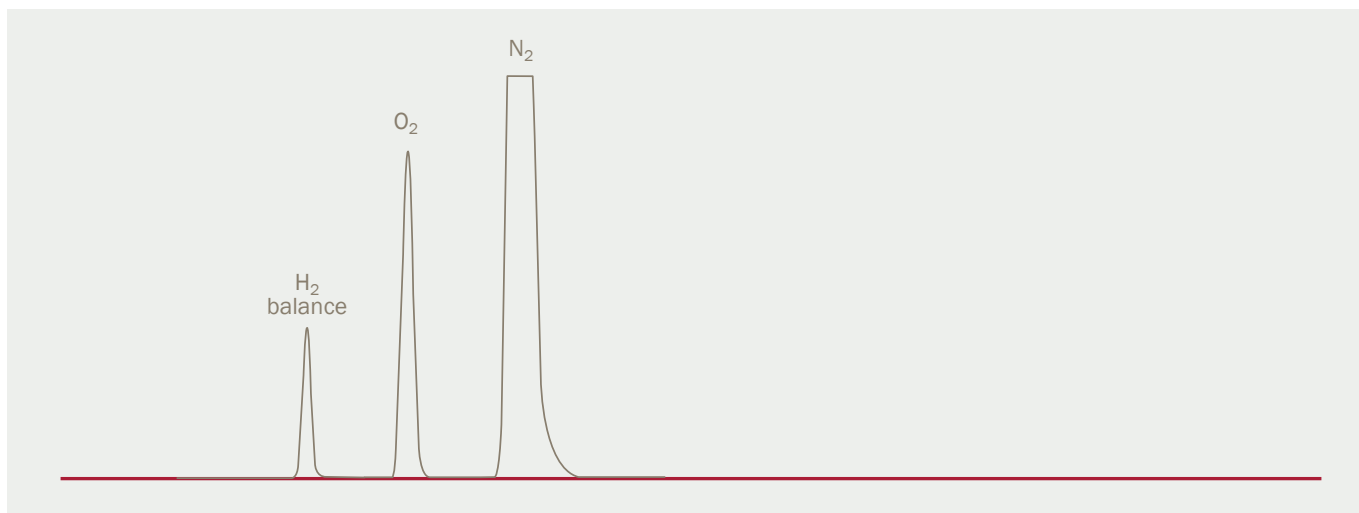


Figure 3 : Chromatogram of traces O₂ - N₂ in a hydrogen matrix using PlasmaDetek-2 in a selective mode.

ARGON AS CARRIER GAS:

Since the PlasmaDetek-2 can be used with argon as carrier gas, you get some more advantages over conventional ionization detector.

Operation cost of the system is reduced compared to system that requires helium. With the helium shortage worldwide and its price increasing constantly, the use of argon becomes the best alternative to keep same sensitivity level.

Furthermore, in the case when argon as impurity is not required, the use of argon as carrier gas simplifies the chromatography. With conventional ionization detector using helium, the Ar will interfere with O₂ measurement. If Ar measurement is needed, the PlasmaDetek-2 can also be used and configured accordingly to get Ar and O₂ separately (see application note LD12-3).

COMPLETE SOLUTION:

Using the PlasmaDetek-2 as detector and the MultiDetek-2 as compact GC platform, the same instrument can be used to measure additional components in hydrogen matrix.

The analysis of traces Ar-Ne-CH₄-CO-CO₂ and hydrocarbons can be added to the same instrument just by configuring additional channels to the compact GC MultiDetek-2. Some other sensors can be added to the same plasma for the added impurities. No needs of additional detectors like FID or consumables are required.

CONCLUSION:

The pay back of such analytical tool is fast. It requires only argon as carrier gas which is available at low cost on any air separation plant. The PlasmaDetek-2 used in the MultiDetek-2 is maintenance free and is a clean detector. It requires no cleaning procedure. The compact GC MultiDetek-2 will operate for many years and gives optimal efficiency of any hydrogen production plant.

The combination of MultiDetek-2 compact GC with the PlasmaDetek-2 technology is a reliable, efficient and accurate system that any plant is looking for.



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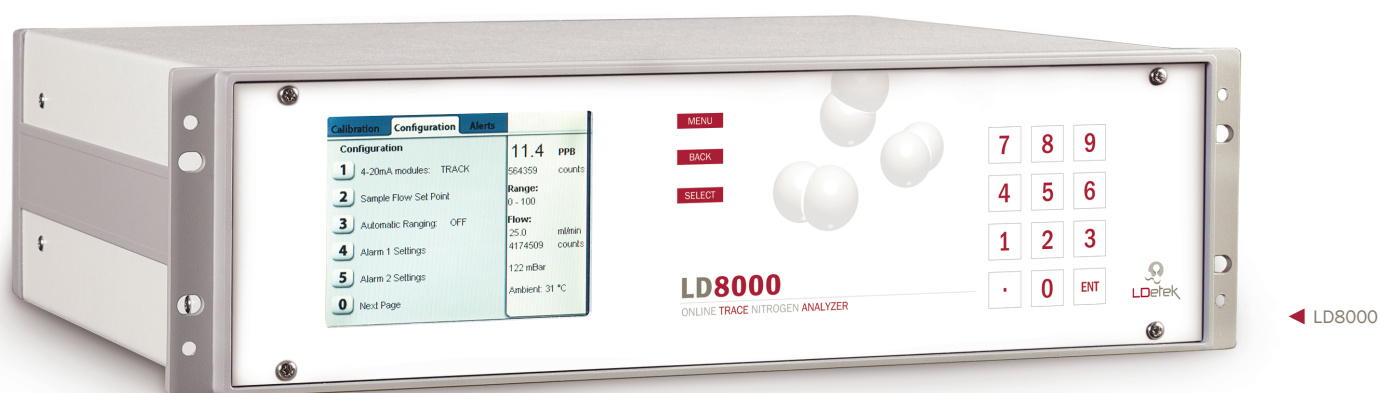
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APPLICATION NOTE

LD13-02



Measurement of nitrogen in a mixture of Argon/Oxygen (crude argon) online with the LD8000-Crude



The measurement online of nitrogen in UHP argon is widely used and the LD8000 is now a reference in such measurement. However when measuring crude argon with a few % of oxygen, the conventional online instruments are not suitable anymore. Such crude liquid argon (also called CLAR) needs to be analyzed in many cases to be sure the level of nitrogen is within the specification either as a final product for steelmaking or welding applications or before being purified to produce UHP argon. Good analytical tool is then required to have a good process control. The use of Gas Chromatograph (GC) has been widely used to measure this nitrogen level. Even if GC is giving good and accurate results, the analysis time is quite long. In many cases the response time is too long to take proper action if the nitrogen goes too high in concentration. A few minutes are required to get the analysis results and this delay can bring a lot of problems to the plant if it increases suddenly.

The need of having an online instrument with a few second response time has been desired for a long time. The LD8000-Crude can now achieve this task and give a better process control for any plant or application that needs to measure N₂ in the Crude Argon having up to 5% oxygen.

DESIGN:

The LD8000-Crude is still using the unique PlasmaDetek technology, but incorporated in a special design, it makes possible to have this plasma emission detector (PED) working with up to 5% oxygen. Common PED cannot create a stable plasma with oxygen. The interference and instability of the plasma is a big challenge. But LDetek has developed a method and a design to have such argon micro-plasma being able to measure from ppb to % of nitrogen with up to 5% oxygen.

A specific algorithm has been developed and implemented in the microcontroller unit giving a linear and accurate measurement of nitrogen.

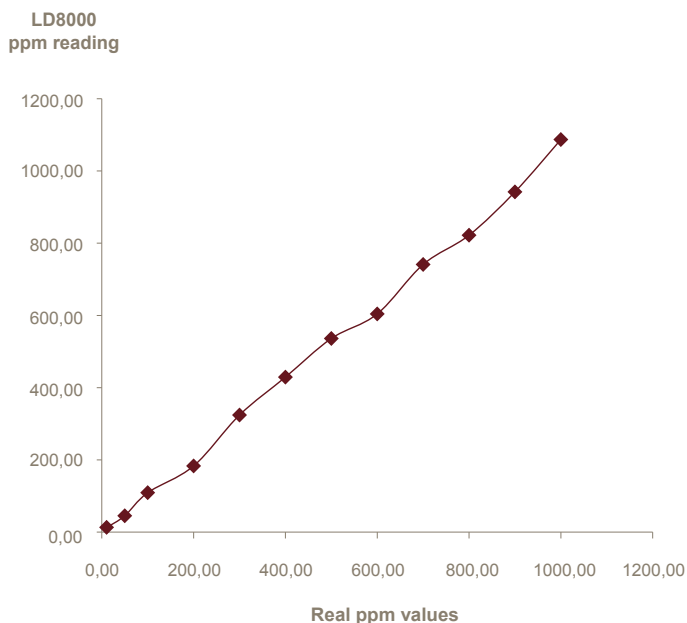


Figure 1 : LD8000 Crude linearity 0-1000 ppm

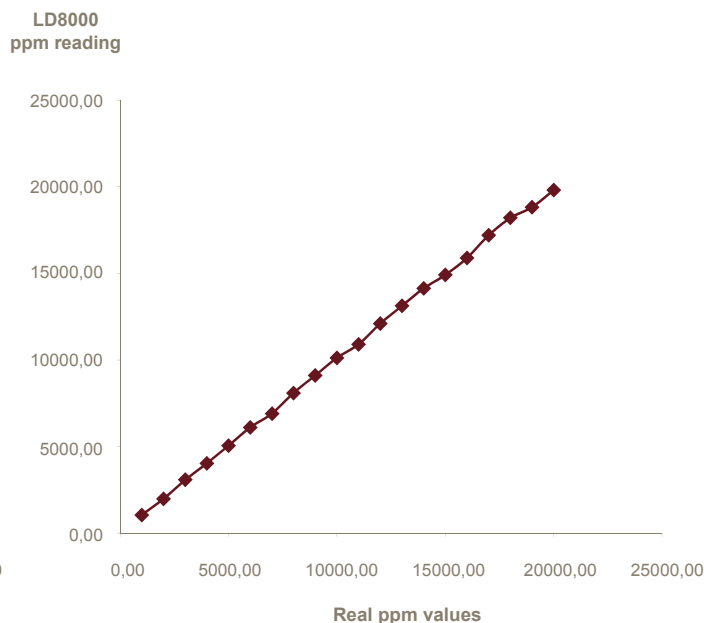


Figure 2 : LD8000 Crude linearity 0-20000 ppm

With the 3U cabinet, this instrument can fit in any rackmount cabinet or plant installation. No need of carrier gas and external consumable parts like purifier or oxygen scrubber. Only sample is needed to provide this quick measurement.

The micro controller unit inside combined with the LCD display provide a user friendly interface easy to operate. But it also gives all features that such instrument needs on a plant (dry contact relay for range ID, status contact for error monitoring, 4-20 mA output for value reading, auto-resolve alarms system, etc).

RESPONSE TIME:

The response time for such application is critical. The use of a gas chromatograph has been widely used and it gives good results, however the analysis time can be as high as 5-6 minutes. It is very important, in most of such applications, to have a quick response time to act quickly on the process when the level of nitrogen goes too high.

Using the LD8000-Crude, you get a response time of a few seconds ($T_{90} < 20$ sec) with a measurement of a few ppm. With the 4-20mA output signal, the process plant can track the level of nitrogen continuously. If serial communication is required, this instrument can send and be controlled by such communication port.

HIGHER OXYGEN CONTENT:

For measurement of N_2 in Crude Argon containing more than 5% Oxygen level, the LD8000-Crude instrument can be configured to achieve such task. The unique plasma detection system (PED) for Crude Argon measurement stays the same. An additional module for sample gas treatment is added to the instrument, which makes the detection of N_2 in Crude Argon having more than 5% Oxygen possible.

For more information and details, do not hesitate to contact LDetek.



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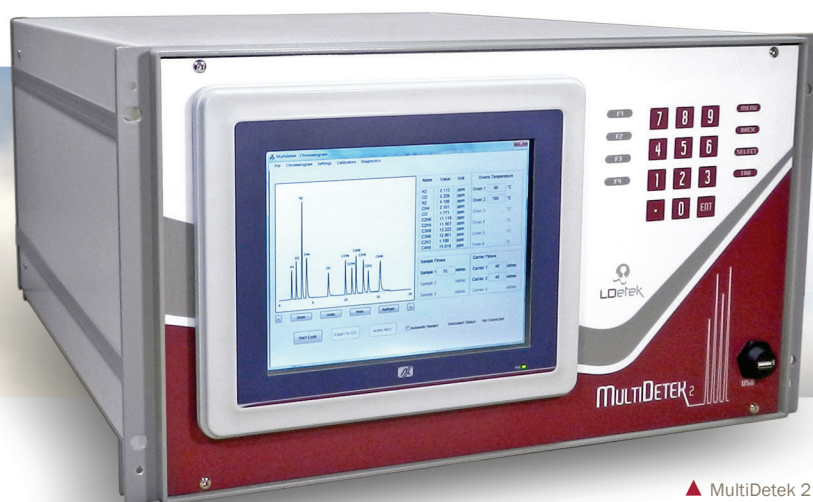
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APPLICATION NOTE

LD13-03



Measurement of H₂S and COS in Syngas with MultiDetek 2



▲ MultiDetek 2

Syngas (Synthesis gas) a fuel gas mixture, primarily composed of hydrogen, carbon monoxide and carbon dioxide, is mainly used as intermediate in creating synthetic natural gas (SNG) or ammonia or methanol.

To be able to use a clean and environmental friendly fuel and feedstock, the sulfurs compounds must be removed. Right analysis tool is needed to ensure that the concentration of sulfurs is kept at the minimum desired level.

SOLUTION:

The MultiDetek 2 (MD2) along with the PlasmaDetek technology bring an ideal and cost effective solution to monitor hydrogen sulfide (H₂S) and carbonyl sulfide (COS) in syngas.

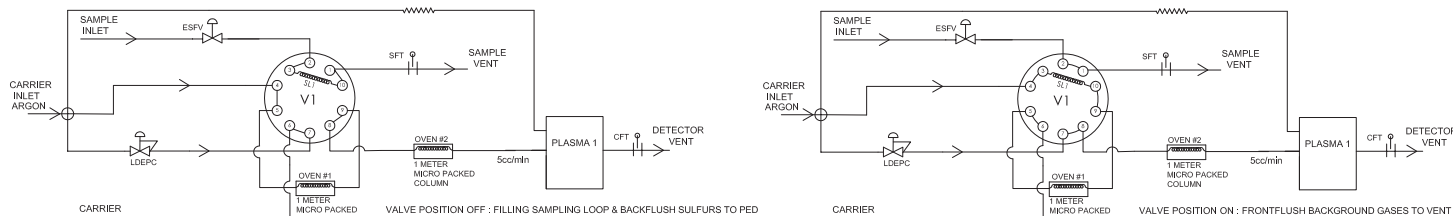
Other common technologies used on the market, like SCD (Sulfur Chemiluminescence Detector) or FPD (Flame Photometric Detector), need hydrogen and air. By using the PlasmaDetek and argon as carrier gas, the MD2 gives a low cost of operation and safer solution. All safety installation for the supply of hydrogen is avoided.



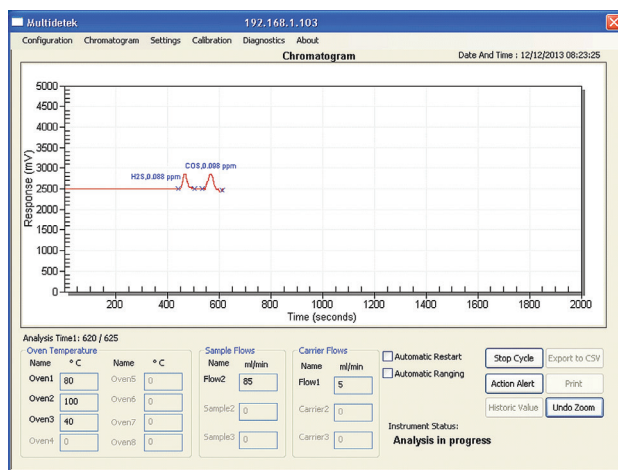
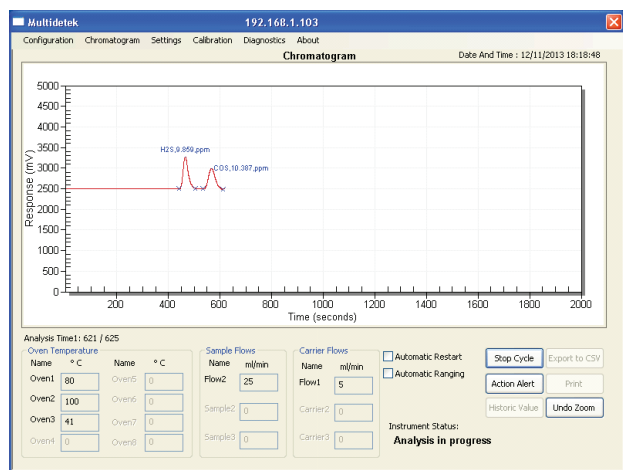
▲ PlasmaDetek

A simple frontflush configuration is used to get rid of the synthesis gas background with the first column. The sulfurs compounds are then well separated by the second column and sent to the PlasmaDetek (PED) especially configured for sulfurs gases. With the use of micro packed type columns, the system can operate with very low carrier flow consumption. The use of argon as carrier gas brings the system even more interesting cause of the low operational cost due to low cost of Argon gas.

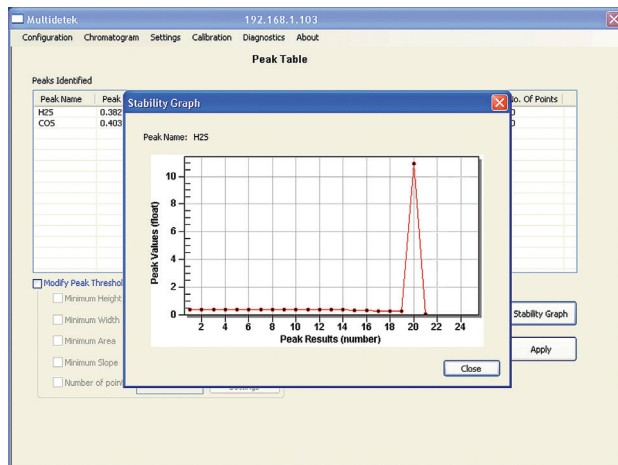
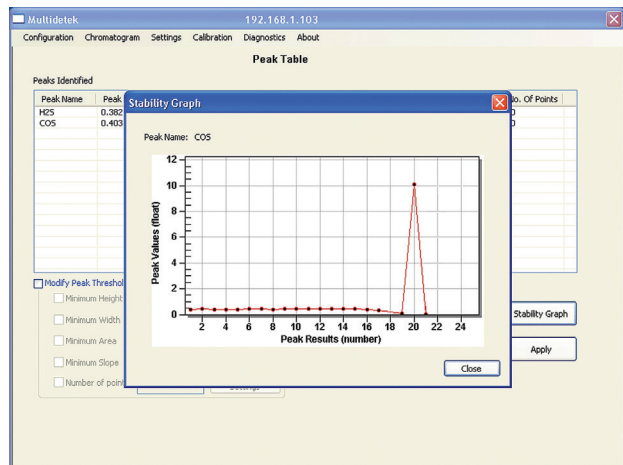
Such MultiDetek operates with column carrier flow of 5sccm. A make up gas of 20sccm is required to the detector to allow good stability and sensitivity. No additional gases or consumables parts are required what minimize the operation and installation cost.



With the combination of the PlasmaDetek and argon carrier gas, detection as low as a few ppb is possible.



Analysis results showing stability on 19 consecutive cycles for H₂S & COS at concentration of 400ppb. Then switch on span gas at 10ppm for H₂S & COS, then switch on zero gas for H₂S & COS.



With the user friendly interface and the configuration of the MD2, it is easy to use the instrument as a process control device or simply a laboratory gas chromatograph to make sporadic analysis.



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APPLICATION NOTE

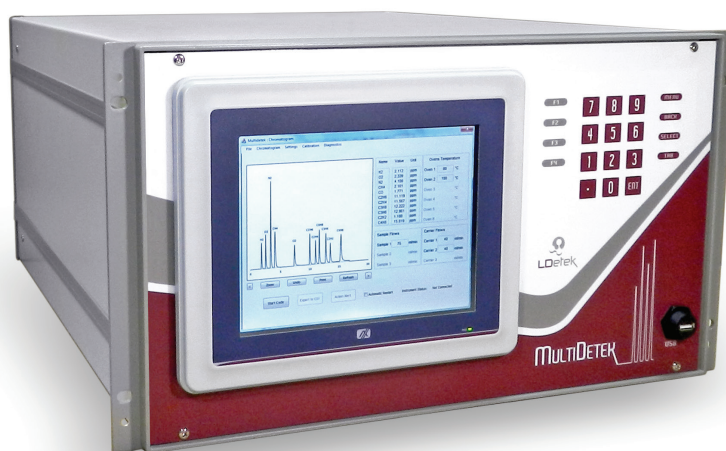
LD14-01



Light hydrocarbons measurement with the *PlasmaDetek-E system with nitrogen carrier gas and the MultiDetek-2.



◀ PlasmaDetek-E



▲ Multidetek-2

Hydrocarbons are ones of the most important impurities measured in the industry. Whether it is for safety, quality control, special gases or any other needs, those compounds are everywhere.

The techniques used to measure those compounds have been the same for quite some years. The FID (Flame Ionization Detector) is surely the most widely used in the industry. The selectivity for hydrocarbon (HC) gives the simplicity desired for all gas chromatograph (GC) users. However the need of air, but mostly hydrogen as fuel is the drawback of this technology. Many plants and laboratories would like to get rid of the hydrogen as potential explosive gas. All the safety feature (valves, extra lines, procedures, etc) are required and brings extra cost and manpower.

Other technologies to measure the light hydrocarbon have arisen over the years. The Discharge Ionization Detector (DID) brought a solution without the need of air and fuel. Working on helium carrier gas, the safety problem was resolved. However the selectivity is not present and the need of helium is required. With the worldwide shortage of helium and its increasing price constantly, it becomes a more costly solution not attractive enough for gas chromatograph developer.

Another solution was brought to the market: the Plasma Emission Detector (PED). With its advantage to work in helium and argon carrier gas, this solution becomes more attractive because of argon pricing. It is also produced and available anywhere in the world. Furthermore, the PED has some selectivity against some other gases, not completely, but enough to make the GC user life easier. However, there is no selectivity for HC against many compounds, including air and oxygen. Measuring HC in a stream of Oxygen or Air is very important in Air Separation Unit (ASU). There is also a lack of sensitivity in some case by using argon as carrier. Measuring C_2H_2 for a ASU plant is critical and it needs to be measured in low concentration.

LDETEK SOLUTION:

With the patent pending PED system (PlasmaDetek-E) provided by LDetek, it is now possible to improve and overcome the drawbacks of other available technologies.

* Patent pending

NITROGEN CARRIER GAS:

First of all, such system can work with argon and helium, but with nitrogen carrier as well. Nitrogen is a low cost and inert gas that makes it very attractive to use in a GC. Air contamination and leak are not much problematic as it is with other PED systems. Typical PED detector can be sensitive, but it needs to be in a very pure environment. A little leak or contamination can compromise the whole measurement. The recovery time from column changes and GC start-up is then extremely fast with the PlasmaDetek-E using nitrogen as carrier gas.

A simple injection configuration has been used to inject different matrices to evaluate the response and selectivity over CnHm components. Figure 1 shows such configuration with the PlasmaDetek-E system.

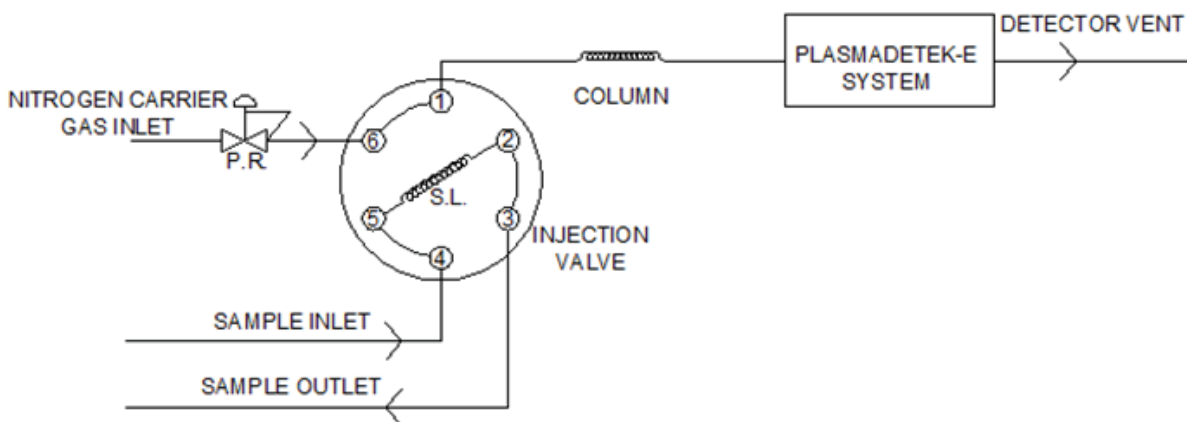


Figure 1

SENSITIVITY:

To evaluate the response and sensitivity, injection of CnHm impurities in Argon balance has been used. Figure 2 shows the chromatogram with indication of each compounds detected by the PlasmaDetek-E.

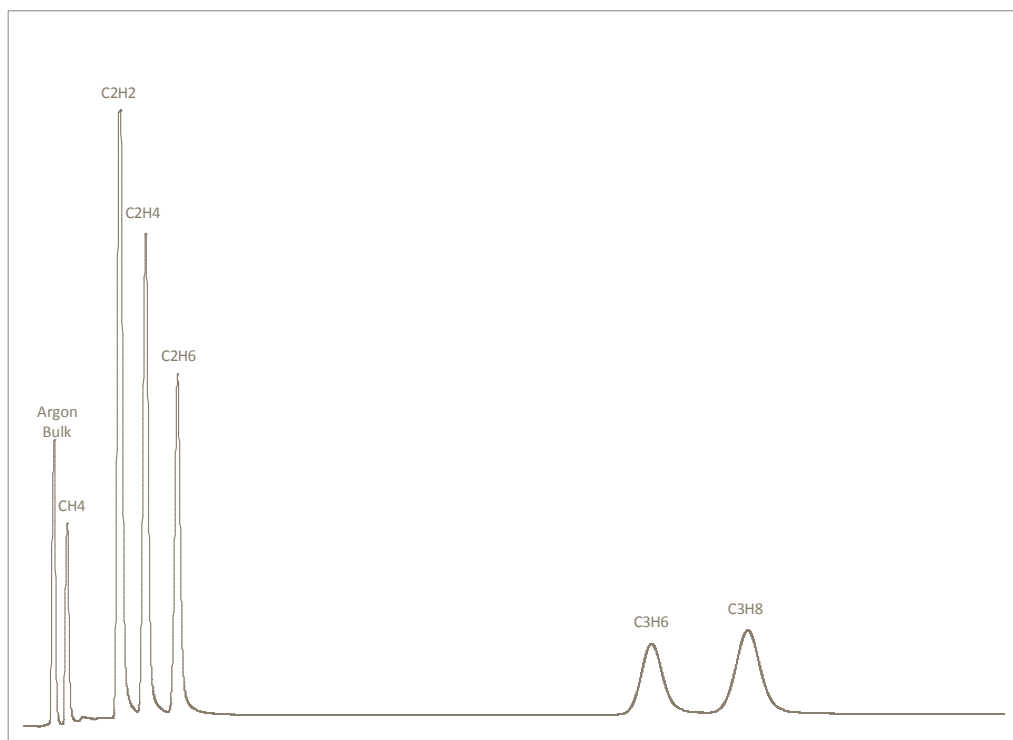


Figure 2

By evaluating the noise level and peak height, LDL for each compounds can be evaluated by considering the limit as 3 times the noise level. Figure 3 is showing the results of this analysis.

COMPONENT	CONCENTRATION	PEAK HEIGHT	NOISE (3x)	LDL (3x NOISE)
CH ₄	9.56 ppm	1300 mV	1.5 mV	11 ppb
C ₂ H ₂	10.50 ppm	4800 mV	1.5 mV	3 ppb
C ₂ H ₄	9.96 ppm	3100 mV	1.5 mV	4 ppb
C ₂ H ₆	10.80 ppm	2300 mV	1.5 mV	7 ppb
C ₃ H ₆	10.40 ppm	460 mV	1.5 mV	33 ppb
C ₃ H ₈	9.20 ppm	560 mV	1.5 mV	24 ppb

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

Figure 3

By using different injection volume and/or chromatographic configuration, even lower LDL could be obtained. However, by comparing with same the chromatographic condition than a traditional argon PED, LDL is improved by a factor of about 10 times with the PlasmaDetek-E. It is even more the case for C₂H₂, where it is about 100 times more sensitive.

SELECTIVITY:

By using the same configuration as figure 1, air has been injected to evaluate the interference that can occur with the first hydrocarbon content out of the column, which is CH₄. Figure 4 shows an injection with the PlasmaDetek-E using argon as carrier gas optimized for the best response to Hydrocarbon in air. It is obvious that the CH₄ is influenced by the residual air. In such cases, extra valves or hardware would be required to achieve possible measurement.

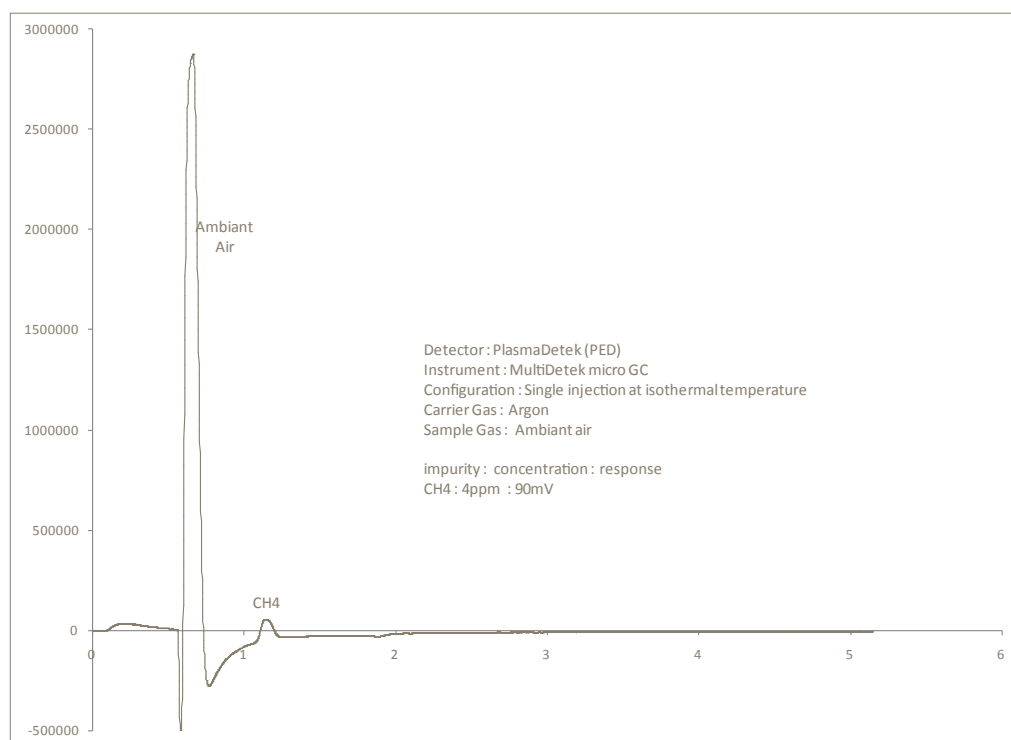


Figure 4

Figure 5 is using the same system with nitrogen carrier gas. Air response has been reduced considerably compared to argon carrier gas. Only with a simple injection configuration will make the CH₄ detectable.

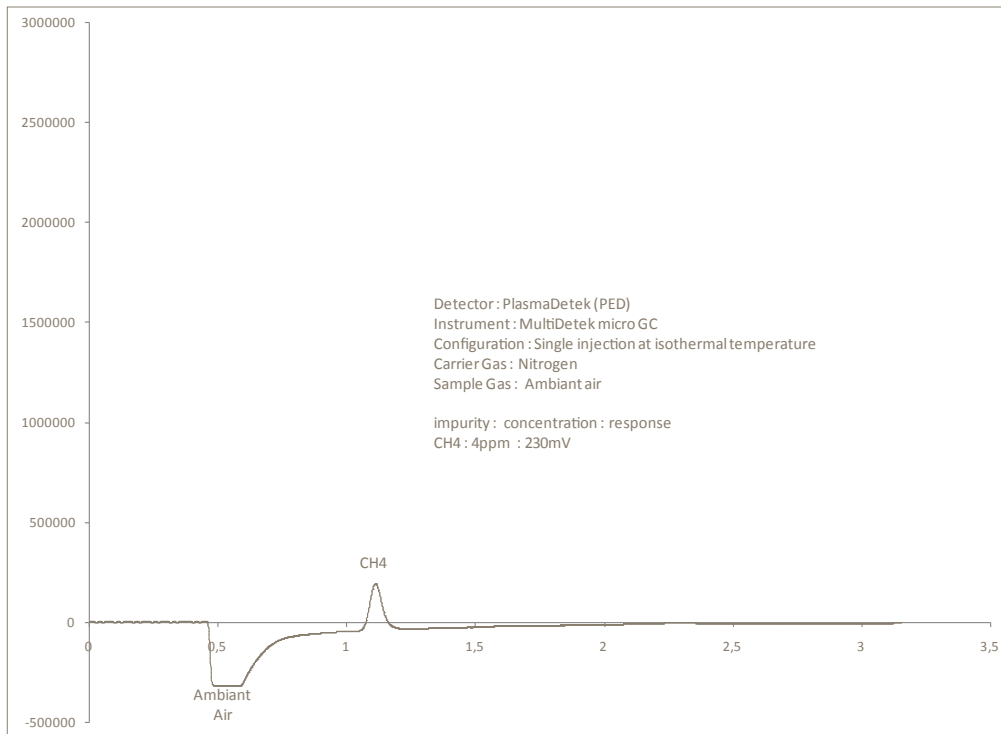


Figure 5

CONCLUSION:

With the PlasmaDetek-E system, lights hydrocarbon measurement is now safe and easy. No fuel, no air and no safety features needed, only nitrogen carrier gas.

It is also the lowest operation cost solution, but the sensitivity is not compromised where low ppb can be achieved.



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APPLICATION NOTE

LD15-02



Measurement of part per billion Ar and N₂ in oxygen for semiconductor industry



▲ MultiDetek-2

The oxidation of silicon is a common and frequent step in the manufacture of integrated circuits (IC). The semiconductor industry requires the production of Ultra High Purity oxygen for the wafer production.

LDETEK SOLUTION:

The oxygen pipeline purity that goes to the semiconductor industry must be properly measured to ensure that there is no contaminant in it. It is critical and challenging to measure the ppb content of argon and nitrogen impurities in oxygen. The conventional technique used for such application is with a heated Oxy-Trap system combined with HID or conventional PED. Such technique requires a complex chromatography system with periodic Oxy-Trap regeneration with hydrogen. The operations of such system require a lot of maintenance and specialist interventions on a routine basis. The limitation of the lifetime of the trap reduces the continuous operation. Depending on the system condition, the Oxy-Trap has to be regenerated more or less once a week.

Our solution consists of eliminating all the consumables and ensuring continuous operation without maintenance and specialist intervention. Moreover, LDetek can also offer an all in one solution using the MultiDeteks-2 combined with the PlasmaDetek-E for monitoring the CO, CO₂ and some hydrocarbons which are critical impurities to measure for semiconductor gases.

Measuring Ar in oxygen :

A first channel using a straight 10 port double injection diaphragm valve V1 combined with the LDetek unique ArgoTek packed column to separate part per billion argon from pure oxygen at an isothermal temperature of 45° Celsius with a carrier flow rate of 20-30sccm. The helium is used as carrier gas source. There is no need of cryogenic or Oxy-Trap system as commonly used. As simple as a basic injection through our ArgoTek packed column to conduct the accurate analysis of argon in oxygen.



* Patent pending

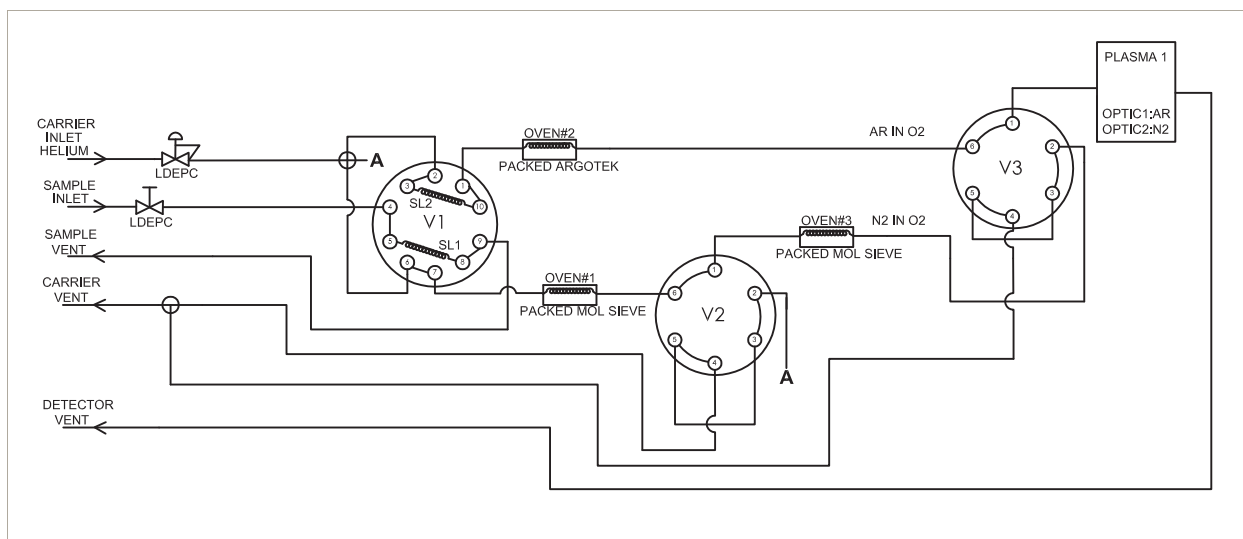
▲ PlasmaDetek-E

Measuring N₂ in oxygen :

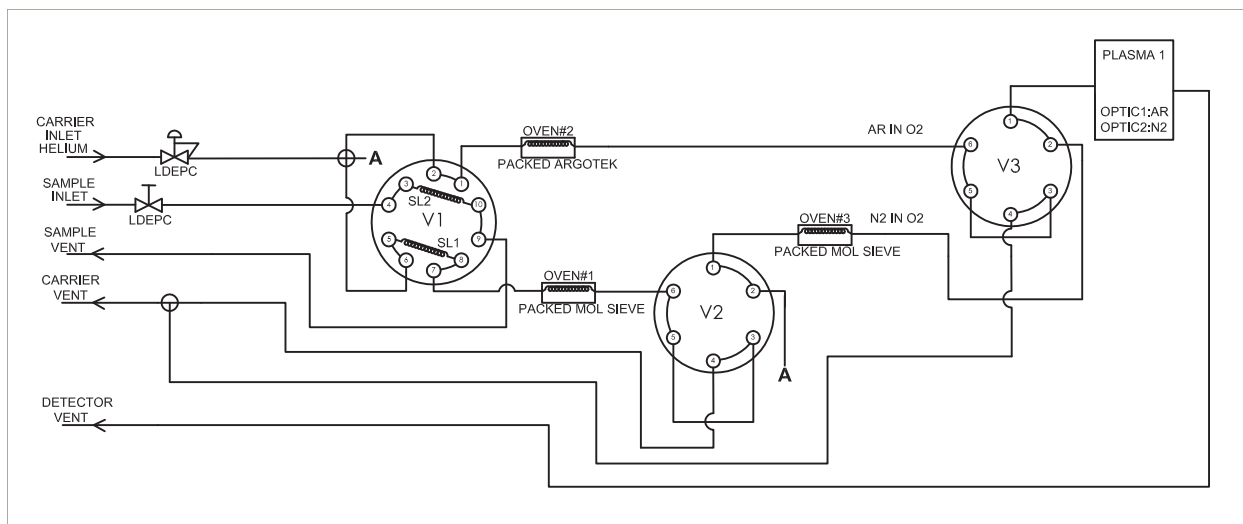
A second channel still using the same straight diaphragm injection valve V1 is used to perform the analysis of N₂ in oxygen with two packed molecular sieve columns combined with a Heartcut diaphragm valve V2. One more time, this configuration allows the measurement of N₂ in oxygen without the use of a complex Oxy-Trap system. Both channels are selected accordingly at the right time with the selection diaphragm valve V3.

PlasmaDetek-E configuration for measuring Ar & N₂ in oxygen :

The PlasmaDetek-E used for this system has a selective configuration to block the interference coming from the oxygen matrix. A specific optic circuit having its wavelength for measuring selectively the argon is mounted in the PlasmaDetek-E. Another specific optic circuit also having its wavelength for selectively measuring the N₂ is in place. The appropriate optic circuit is automatically selected at the proper elution time to allow peak integration. The selectivity of the PlasmaDetek-2 for the Ar-N₂ in oxygen gives the ability to have an easy to use system allowing quick analysis time even at ultra low concentration.



MultiDetek-2 configuration diagram #1 » V1 position OFF: Filling loop #1 and injecting loop #2
V2 position OFF: Catch N₂ peak from the first Packed Mol Sieve in Oven #1 to the second Packed Mol Sieve in Oven #2
V3 position OFF: Select channel #2 for Ar in O₂ to the PED



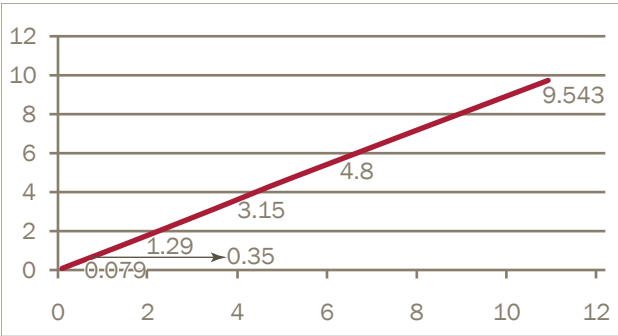
MultiDetek-2 configuration diagram #2 » V1 position ON: Injecting loop #1 and filling loop #2
V2 position ON: Venting oxygen out of the system
V3 position ON: Select channel #1 for N₂ in O₂ to the PED

RESULTS:

Linearity on a 0-10ppm range

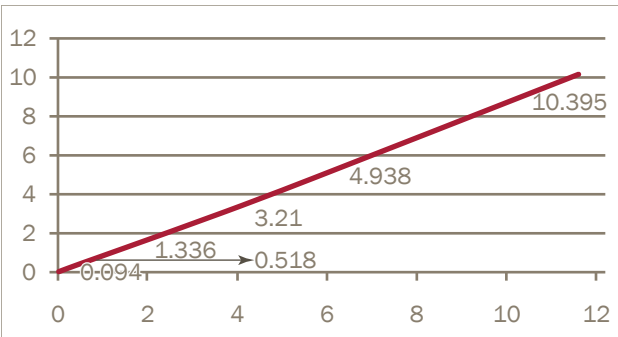
Argon impurity

DILUTED GAS (PPM)	CALCULATED VALUE (PPM)
0.081	0.079
0.35	0.31
1.3	1.29
3.13	3.15
4.73	4.8
9.6	9.543

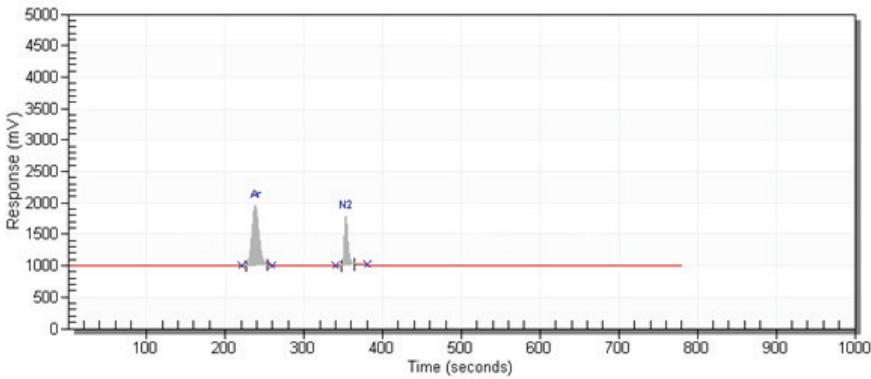


Nitrogen impurity

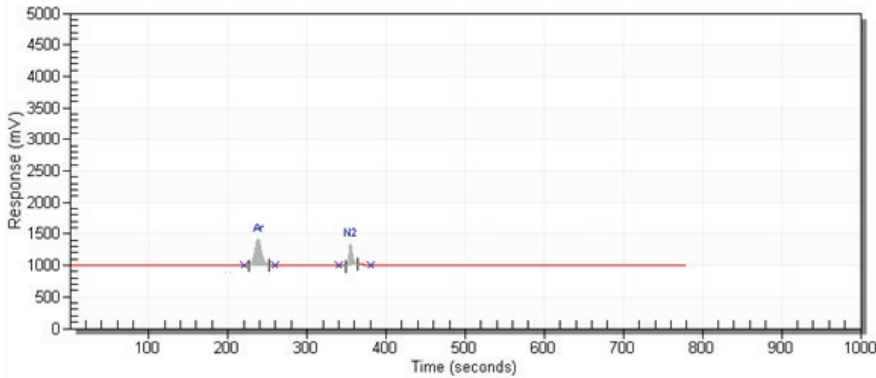
DILUTED GAS (PPM)	CALCULATED VALUE (PPM)
0.097	0.094
0.53	0.518
1.4	1.336
3.36	3.21
5.07	4.938
10.3	10.395



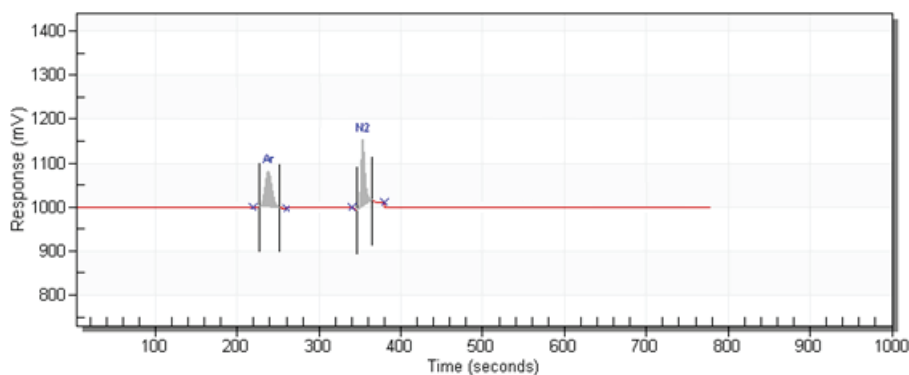
LOW PPB CHROMATOGRAM EXAMPLES:



Chromatogram of 61ppb Ar & 77ppb N₂ in pure oxygen



Chromatogram of 30ppb Ar & 38ppb N₂ in pure oxygen



Chromatogram of 3ppb Ar & 3.8ppb N₂ in pure oxygen

LDL calculation

COMPONENT	CONCENTRATION	PEAK HEIGHT	NOISE	LDL (3X NOISE)
Ar	3ppb	95 mV	1.5 mV	0.1ppb
N ₂	3.8ppb	156 mV	2.7 mV	0.2ppb

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

CONCLUSION:

With its user friendly interface and the simple configuration of this compact MultiDetek-2 GC, it is easy to use the instrument as a process control analyser that is a perfect fit for this semiconductor application. The enhanced sensitivity and selectivity of the PlasmaDetek-E allows extreme low limit of detection. Combined with the LDetek exclusive ArgoTek column, it makes this system perfectly suitable for semiconductor industry.



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APPLICATION NOTE

LD15-03



Measurement of part per billion N₂O in air



▲ MultiDetek-2

With the global warming concerns, it is more and more critical to measure the nitrous oxide (N₂O) concentration in the ambient air. This application note will demonstrate how efficient the use of the PlasmaDetek-E is for the measurement of extremely low concentration of N₂O in ambient air without interference.

LDETEK SOLUTION:

It is well known that the measurement of N₂O can be achieved with different kinds of detectors using specific chromatography techniques. The ECD, HID and conventional PED are the mostly used type of detectors for this greenhouse application. The radioactive source coming from the ECD makes this detector less attractive due to the latest worldwide regulations about radioactivity, even for low source of radioactivity. The HID and conventional PED are relatively good detectors for such analysis, but create more chromatography challenges caused by lack of selectivity and sensitivity. With the LDetek PlasmaDetek-E, the enhanced sensitivity and selectivity make it the ideal tool for measuring extremely low concentration N₂O in ambient air with reduced interference mostly coming from the high concentration carbon dioxide and moisture.

The improved operation's mode and the optical design of the PlasmaDetek-E combined with the right chromatography configuration in the MultiDetek-2 compact GC remove the interference usually coming from the carbon dioxide and moisture.

The MultiDetek-2 system is configured with one injection diaphragm valve V1, which is used for injecting the sampling volume into the chromatography system. A first packed Shincarbon column is combined with a heartcut diaphragm valve V2 and a second packed Shincarbon column to catch the desired N₂O peak and flush to vent the undesired interference gases. The N₂O peak is then going to the PlasmaDetek-E where proper analysis is performed.



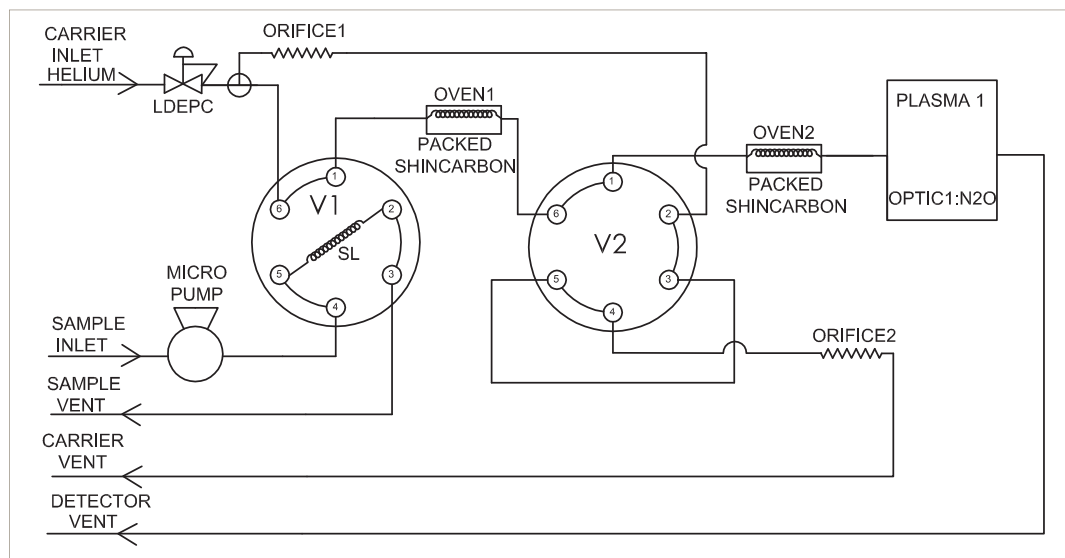
▲ PlasmaDetek-E

This combination makes it ideal to measure extremely low concentration N_2O in air without having the N_2O peak integrated in the high concentration CO_2 tailing. This technique has been tested up to 5000ppm CO_2 for measuring as low as 1ppb N_2O as demonstrated in the chromatograms.

The MultiDetek-2 can be configured with different sampling mode to get the sample gas ready for analysis. The drawing of this application note shows the use of a micro pump that is mounted just before the sampling loop. The pump is fully controlled from the software interface and can be switched On/Off adequately for a specific period of time to allow proper sample gas quantity going to the sampling loop mounted on the diaphragm valve V1. This way, the loop is filled just before running an analysis. This technique allows to minimize the sample gas volume required for analysis since in many cases, the sample gas comes from bags, balloons, canisters or others. It is then critical to minimize the sample gas volume for every analysis.

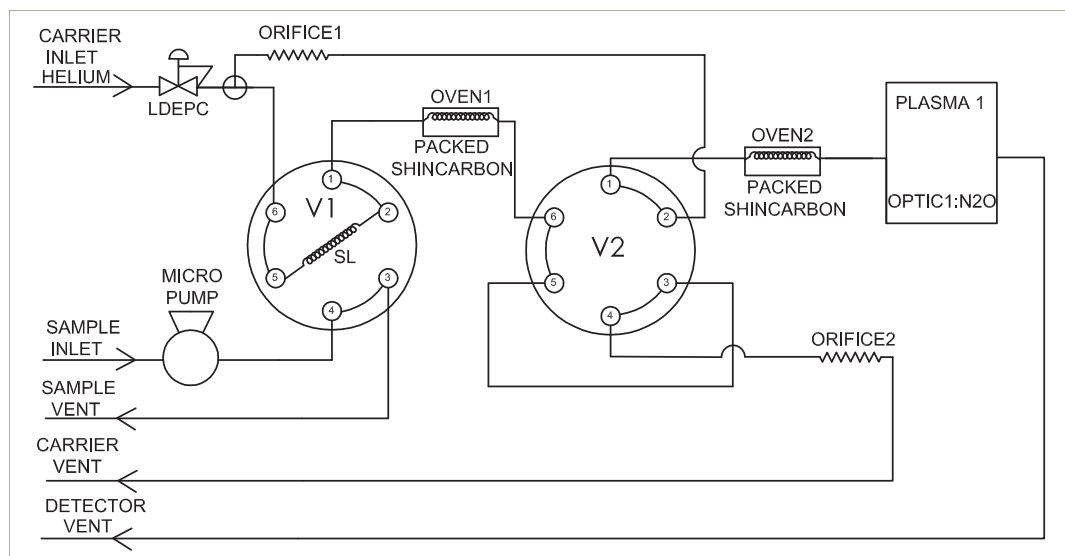
An optional sample line purge system can also be added to the sampling mode of the MultiDetek-2. This system uses the carrier gas to purge the sample line prior to pump the sample gas from its source. This way, the sample flow path and sampling loop are totally purged with N_2O free gas since the carrier gas used as purge gas comes from the heated LDetek, model LDP1000 getter that removes N_2O down to 0.01ppb level.

The MultiDetek-2 can also have an injector system with septum for syringe injection mode. The system is directly mounted on the injection diaphragm valve V1.



MultiDetek-2 configuration diagram #1 » V1 position OFF: Filling sampling loop

V2 position OFF: Catches N_2 peak from the first packed Shincarbon in oven #1 to the second packed Shincarbon in oven #2

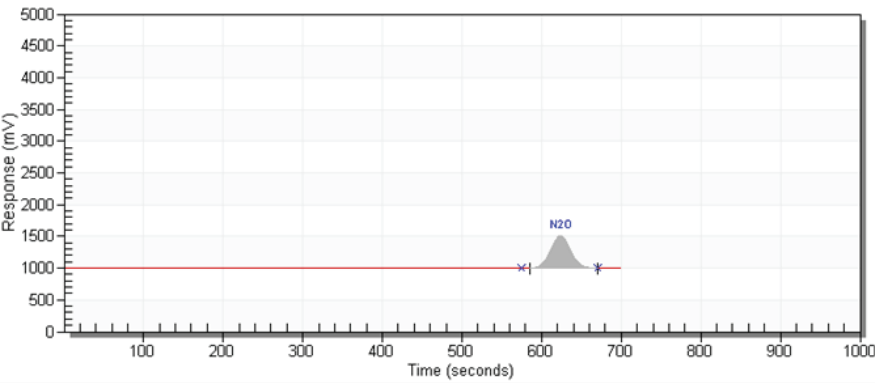


MultiDetek-2 configuration diagram #2 » V1 position ON: Injecting sampling loop

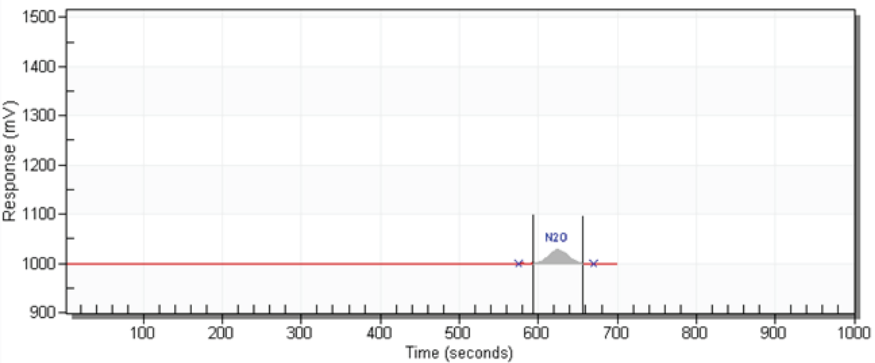
V2 position ON: Flushes to vent the interference gases (air, carbon dioxide, moisture and others) coming out from the first packed Shincarbon in oven #1

RESULTS:

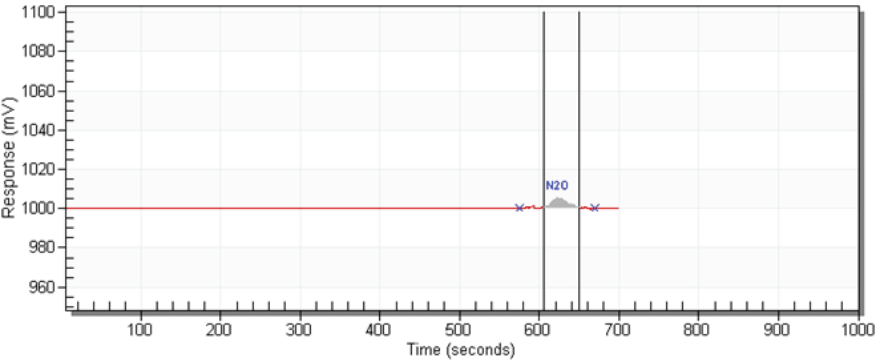
Low ppb Chromatogram examples



Chromatogram of 140ppb N₂O in air



Chromatogram of 7ppb N₂O in air



Chromatogram of 1ppb N₂O in air

LDL calculation

COMPONENT	CONCENTRATION	PEAK HEIGHT	NOISE	LDL (3X NOISE)
N ₂ O	1ppb	6 mV	0.2 mV	0.1ppb

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

CONCLUSION:

With its user-friendly interface and the simple configuration of this compact GC, the MultiDetek-2 is a perfect fit for this greenhouse application. The enhanced sensitivity and selectivity of the PlasmaDetek-E allow extreme low limits of detection. This new measurement technique combined with the integrated purged low volume sampling gas system brings this technology over the existing conventional measurement methods and systems.



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APPLICATION NOTE

LD15-04



Measurement of silane purity in electronic gases industry using PlasmaDetek-E and MultiDetek2



Silane (SiH_4), more properly known as monosilane and alternately called silicon tetrahydride or silicane, is a highly flammable and hazardous chemical compound containing silicon (87.45%) and hydrogen (12.55%). With silicon comprising 87.45% of its content by weight, pure silane is a primary source of high-purity silicon for use in industry. It is a critical gas in the manufacture of semiconductor devices, display panels and other electronic devices. The analysis of ultra low part per billion of permanent gases in silane is required for measuring the silane purity. The use of the LDetek compact & industrial MultiDetek2 GC combined with the PlasmaDetek-E is the perfect fit for this domain of application.

LDETEK SOLUTION:

Handling highly flammable gases like silane requires a high level of safety and this is what LDetek offers with its built-in sample purging and monitoring system inside the MultiDetek2 compact GC. This system consists of 4 steps of safety:

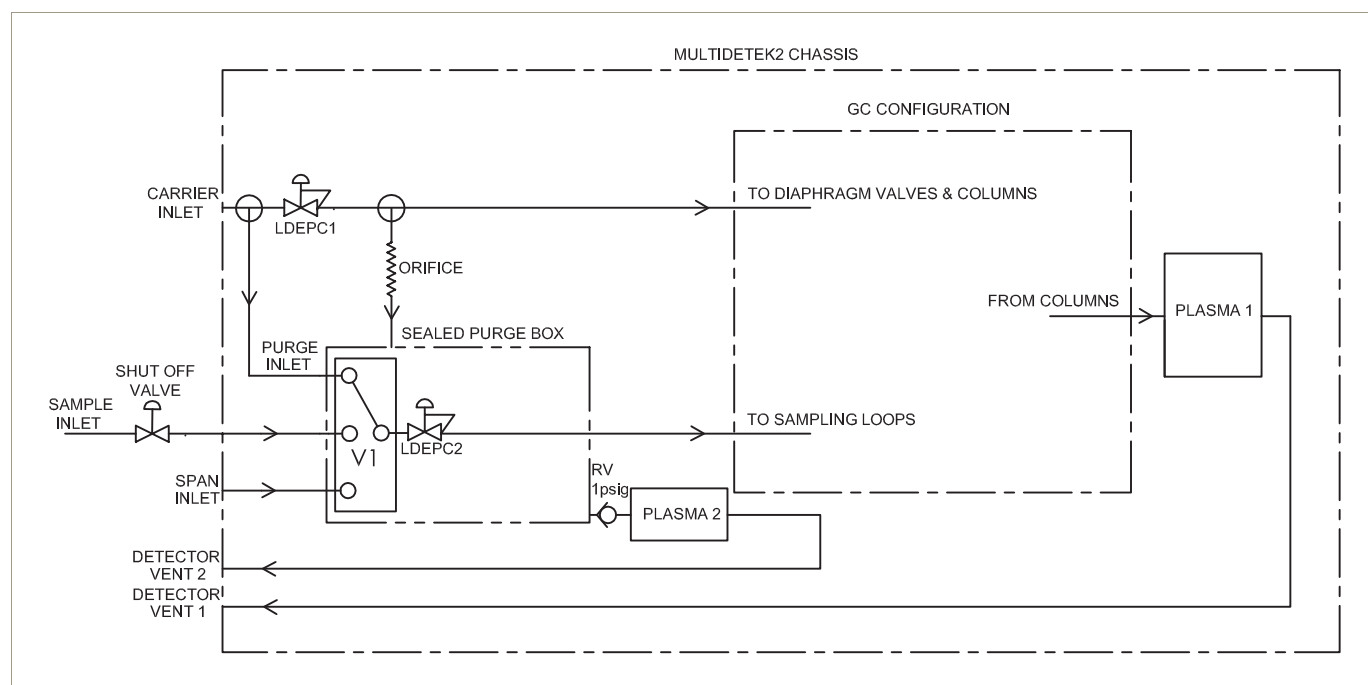
Step 1: The sample gas flow path external to the purge box is fully welded offering no possibility of leakage.

Step 2: A selection valve (V1) is mounted before the diaphragm valves used for filling the sampling loops in the system. That selection valve (V1) is configured to allow silane to go to the sampling loops only for a predetermined period of time, just before the start of each

analysis. The short period of time is configured in the factory and depends on the system configuration. The rest of the time, the selection valve (V1) is switched to a purge gas, which is normally the same gas type as the carrier gas of the system for purging the sampling loops. In the eventuality of leakage on the sampling loops or on the injection diaphragm valves, the reduced period of time introducing silane in the injection valves combined with the low pressure operation and small volume will avoid any potential hazardous situation.

Step 3: A sealed purge box containing the sample flow electronic pressure controller (LDEPC2) and the diaphragm selection valve (V1) is mounted in the MultiDetek2 chassis. That box is normally purged with the same gas type as the carrier gas used for the system. That environment requires low purge flow rate of about 10-30 sccm through a fixed orifice depending on the system configuration. The box is fully ambient air free and the system is ready to use after a short period of about 20-30 minutes depending on the system configuration. This waiting period is only required for initial start-up since once the MultiDetek2 has carrier flow going in it, the box is continuously on purge. Having a box fully purged with UHP carrier gas eliminates the ignition risk in potential presence of silane. A 1psig relief valve (RV) is mounted on the box to build up a minimum sealing pressure and to maintain a constant purge with the carrier gas.

Step 4: The sealed purge box is continuously monitored using a micro PED (PLASMA2) to selectively measure trace N2 to ensure there is no air contamination in the sealed box to avoid the ignition in the potential presence of silane. If trace of air is measured in the purge box by the micro PED (PLASMA2), then an alarm is activated to shut off the flow of silane gas inside the MultiDetek2. The shut-off valve must be mounted external to the MultiDetek2 chassis. The feedback signal controlling the shut off valve comes from the MultiDetek2. It is required to maintain the sample gas pressure coming to the shut-off valve below 10psig to minimize the risk of ignition.



The flow type selected by the selection valve (V1) is controlled with the electronic flow controller (LDEPC2) to ensure a stable and constant flow rate whichever the selected gas type.

The GC configuration for this application is configured with Stainless Steel purge diaphragm valves and MXT column types. At the end of the GC configuration, a PlasmaDetek-E detector is configured to selectively measure the impurities requested. Multiple channels can be configured depending on the application. For this application note, the analysis of H_2 - O_2 - N_2 - CH_4 - CO is measured through channel #1 and the CO_2 through channel#2. Both channels are combined together and go in the PlasmaDetek2 detector. The highly sensitive PlasmaDetek-E allows good detection limit, what is required for silane purity.

RESULTS:

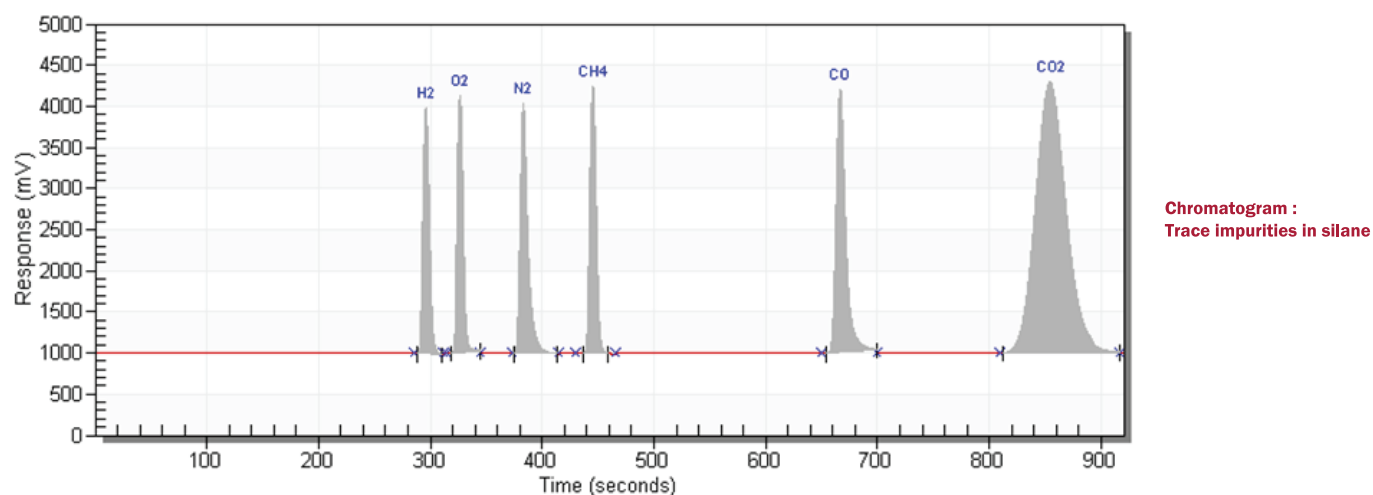


Chart : LDL calculation

COMPONENT	CONCENTRATION	PEAK HEIGHT	NOISE	LDL (3X NOISE)
H ₂	808ppb	2995mV	3mV	2.5ppb
O ₂	810ppb	3220mV	3mV	2ppb
N ₂	810ppb	3095mV	2mV	1.5ppb
CH ₄	810ppb	3335mV	3mV	2ppb
CO	820ppb	3297mV	4mV	3ppb
CO ₂	820ppb	3380mV	3mV	2ppb

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

CONCLUSION:

The PlasmaDetek-E and the MultiDetek2 combined with the highly safe continuous monitoring sampling system allow the measurement of silane purity with reduced risk. The N₂ monitoring of the purge box is essential to ensure there is no ignition risk inside the MultiDetek2.

The measurement of trace of impurities can be performed with a relatively short analysis time, and can offer very low limit of detection what is required for silane purity.



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APPLICATION NOTE

LD15-08



Measurement of impurities in UHP helium using MultiDetek2 and PlasmaDetek2



▲ MultiDetek2

▲ PlasmaDetek2

Helium is a widely used gas in different needs such as cryogenics, pressurizing and purging, welding, controlled atmospheres, leak detection and breathing mixtures. Having a good analytical tool is mandatory to ensure the required purity of helium.

The most popular technique for UHP helium analysis is to detect impurities by gas chromatography. But some detection technologies within the GC do not provide the desired detection limit or can simply not measure some critical impurities like neon.

LDETEK SOLUTION:

The MultiDetek2 combined with the PlasmaDetek2 detector provides an ideal solution to measure the different impurities in UHP helium. With the PlasmaDetek2, based on plasma emission detection, impurities in low ppb can be detected, even neon. The discharge ionisation detector (DID) can simply not detect neon or require a different operation mode to achieve ppm measurement. Which is not enough for many helium producers where they need lower detection limit in ppb. The operation is also more complex since the ionisation voltage needs to be changed when neon has to be detected.

LDetek solution gives an easy system to use with very good sensitivity. Figure 1 shows such configuration.

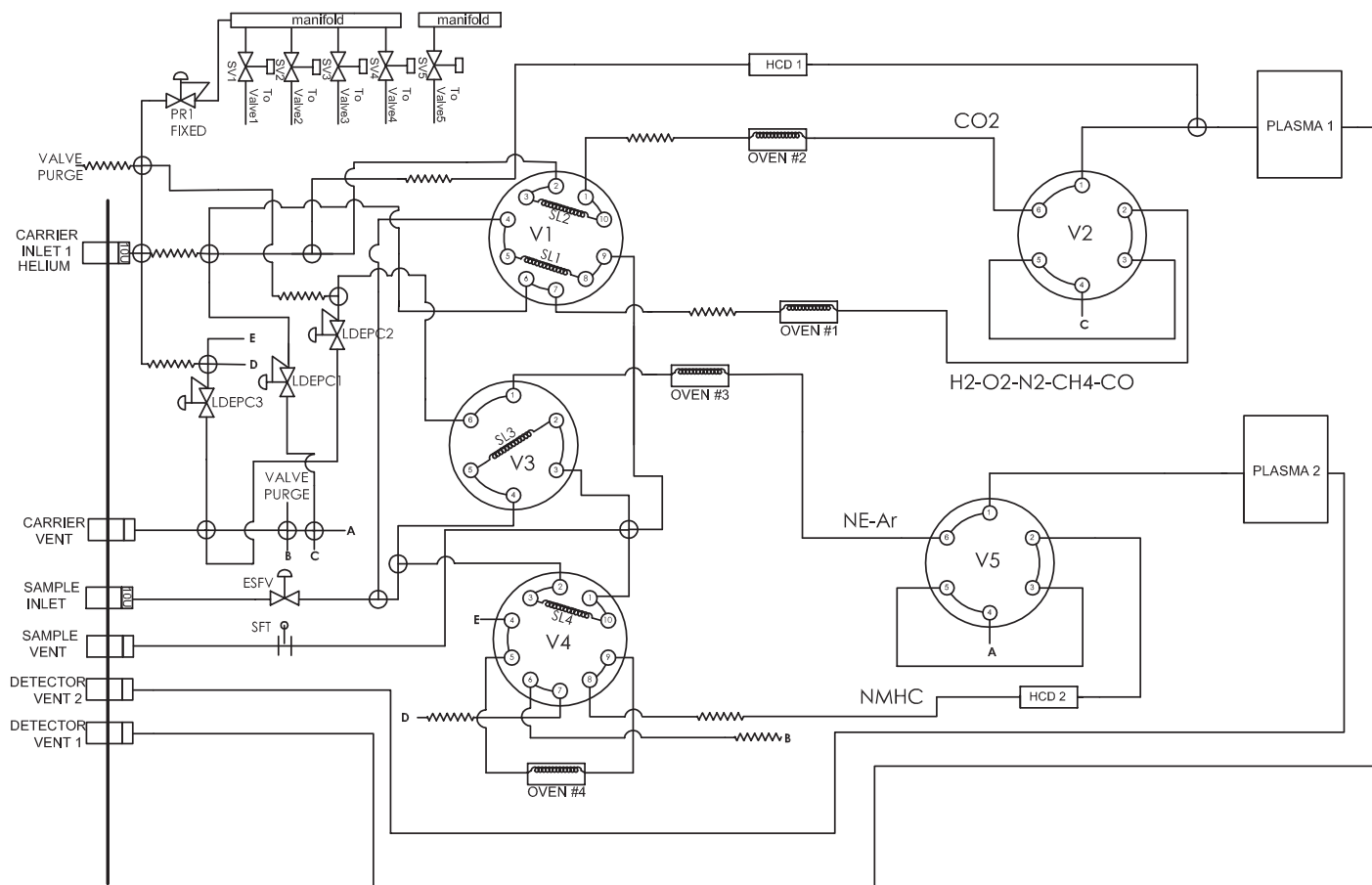


Figure 1

Two plasma cells are used to reduce the number of selection valves. Simultaneous injection in both plasmas is possible to accelerate analysis time. The parallel analysis feature within the MultiDetek2 software can be enabled to give the opportunity to acquire two chromatograms at the same time.

Neon and argon are measured within the same channel using the argotek column from LDetek. Such column gives a true argon peak by separating O₂ and argon completely. Be sure to check application note LD12-3 on our website for more details. It also makes possible the measurement of neon against H₂.

When using the PlasmaDetek2 technology, different optical filters are used to ensure true measurement of the components when interference can occur. For example, O₂ is measured accurately against argon by using a specific optical filter giving a signal specifically to O₂ and not argon.

figure 2 shows a chromatogram of such system with a standard gas.

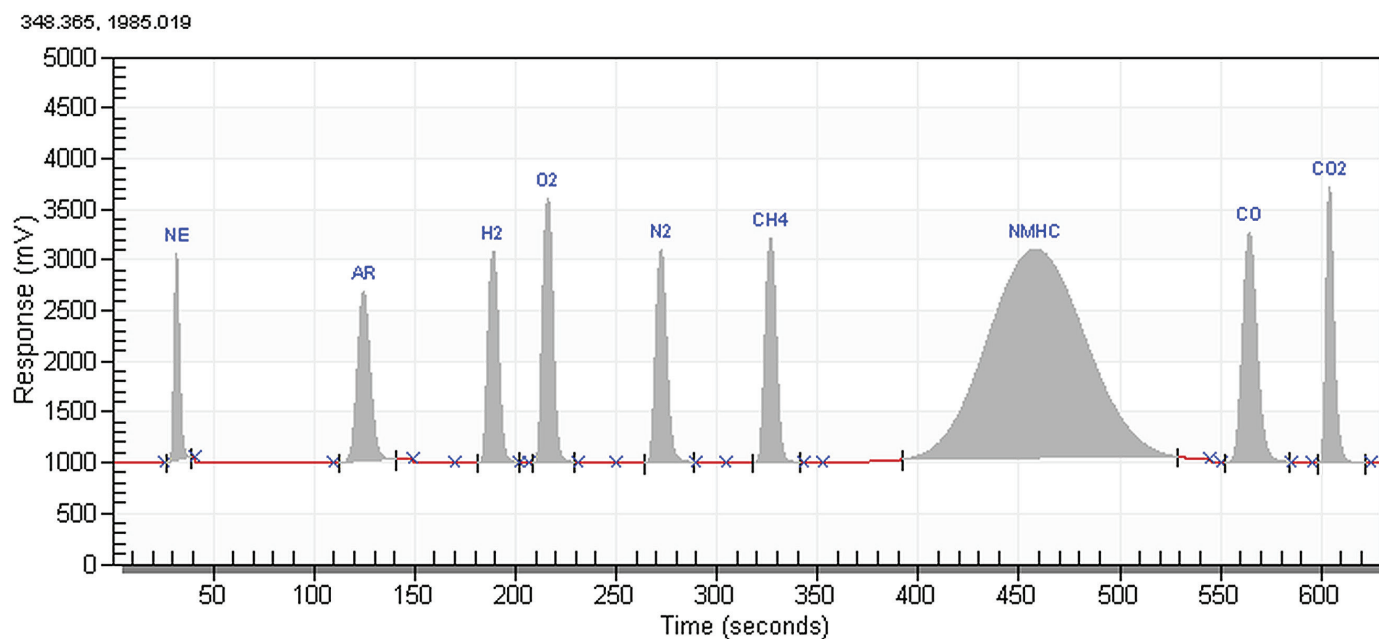


Figure 2

Based on noise to ratio, LDL is calculated as follows:

COMPONENT	CONCENTRATION	PEAK HEIGHT	NOISE	LDL (3X NOISE)
Ne	4.8 ppm	3010 mV	2 mv	0.009 ppm
Ar	4.1 ppm	2720 mV	2 mv	0.009 ppm
H ₂	5.5 ppm	3120 mV	2 mv	0.011 ppm
O ₂	6.2 ppm	3552 mV	2 mv	0.010 ppm
N ₂	5.4 ppm	3099 mV	2 mv	0.010 ppm
CH ₄	5.5 ppm	3254 mV	2 mv	0.010 ppm
NMHC	4.6 ppm	3101 mV	3 mv	0.007 ppm
CO	5.4 ppm	3331 mV	2 mv	0.013 ppm

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

COMPLETE SOLUTION:

Our Helium purity analysis offers a complete cabinet solution including the MultiDetek2 compact GC system, the LDGSS ultra high purity remote stream selector system, the LDP1000 carrier gas purifier and the moisture analysis instrument all integrated in one cabinet. The stream selector can be manually controlled using the front switch or can be remotely controlled with the MultiDetek2. A pre programmed sequence can also be done with LDGSS. The moisture analysis is re-directed to the MultiDetek2 analog input. This way, all data can be managed by MultiDetek2 using one channel communication protocol. The system is pre-configured and certified by experts at LDetek facility before shipping.



CONCLUSION:

Using both the PlasmaDetek2 and the MultiDetek2, only one system can measure all permanent gases with the sensitivity required. No need of additional oxygen analyzer or other setup for neon measurement. Furthermore, with the LDetek technology this maintenance free system avoids any trap or scrubber for the argon impurity.



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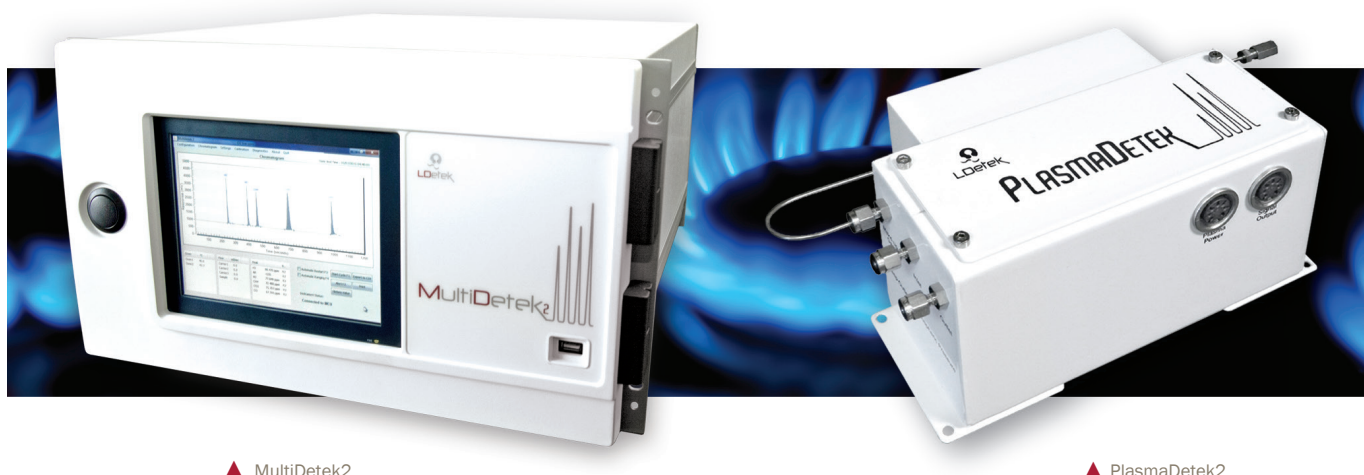
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APPLICATION NOTE

LD15-09



Measurement of THT in natural gas using MultiDetek2 and PlasmaDetek2



▲ MultiDetek2

▲ PlasmaDetek2

A quick analysis to trace tetrahydrothiophene (THT) in natural gas is required for controlling the amount of this odorant added in the natural gas. Due to its odor, the THT is used to detect any presence of gas leakage in natural gas distribution networks. The THT is composed of a five-membered ring containing four carbon atoms and a sulfur atom. It is also known as thiophane or thiolane. The THT is generally used in mixtures containing tert-Butylthiol (TBM) which is an organosulfur compound with the formula $(CH_3)_3CSH$. In the presence of TBM in natural gas, it is required to measure its concentration at low ppb/ppm because of its strong odor that causes nausea. The permissible exposure limit (PEL) is in the range of 500ppb and it is the reason why a highly sensitive detection system is required for measuring both THT and TBM in natural gas.

LDETEK SOLUTION:

The use of a highly sensitive detection system (PlasmaDetek2) combined with a compact GC system (MultiDetek2) are required for such type of analysis where sensitivity, robustness and speed are necessary. The figure 1 shows the easy to operate 2-valve/2-column configuration used to achieve a quick analysis of THT at low ppb/ppm concentration with no interference from other gases present in natural gas. This type of configuration is straight and ensures a good stability of the measurement over time. The valves, tubing, fittings are made of coated stainless steel to ensure repeatability and durability of the system. The robustness of the injection and detection system makes this solution maintenance free on long-term operation.

According to the minimum detection limit that is required, the analysis time can vary. The figure 2 shows a chromatogram of such system having a limit of detection fixed at 31ppb. Analysis time is then fixed below 4 minutes, what is the average time generally required for this type of measurement. If quicker analysis time is requested, the system parameters can be easily modified to achieve lower cycle time analysis. That generally consists of sampling loop volume and system gain reduction to achieve a higher ppb detection limit.

The PED detection system can be configured either with Helium or Argon as carrier gas depending of the sensitivity requested. It is a unique carrier gas source with low gas consumption for cost saving.

The analysis of tert-Butylthiol (TBM) can be added in the same MultiDetek2 compact GC still using PlasmaDetek2 (PED) as the detection system.

With its built in industrial PC, the MultiDetek2 offers all the conventional communication protocols (analog output, Modbus, Profibus, RS232), data storage disk and alarms contacts required for a process GC.

Figure 1 : Application hardware configuration

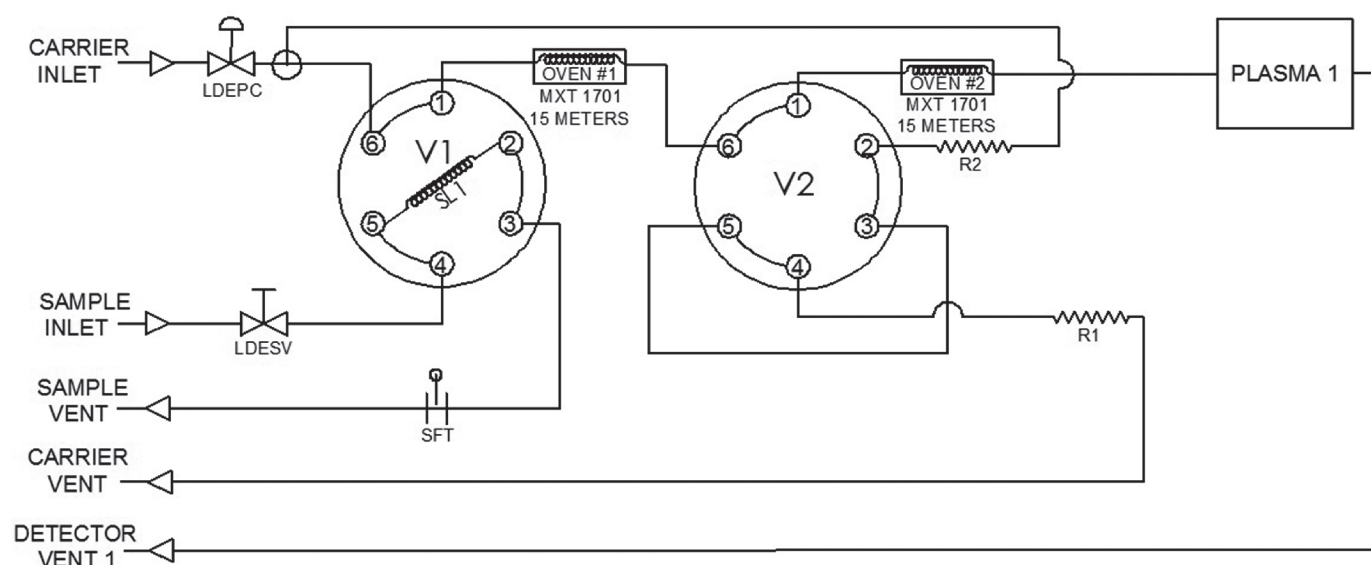
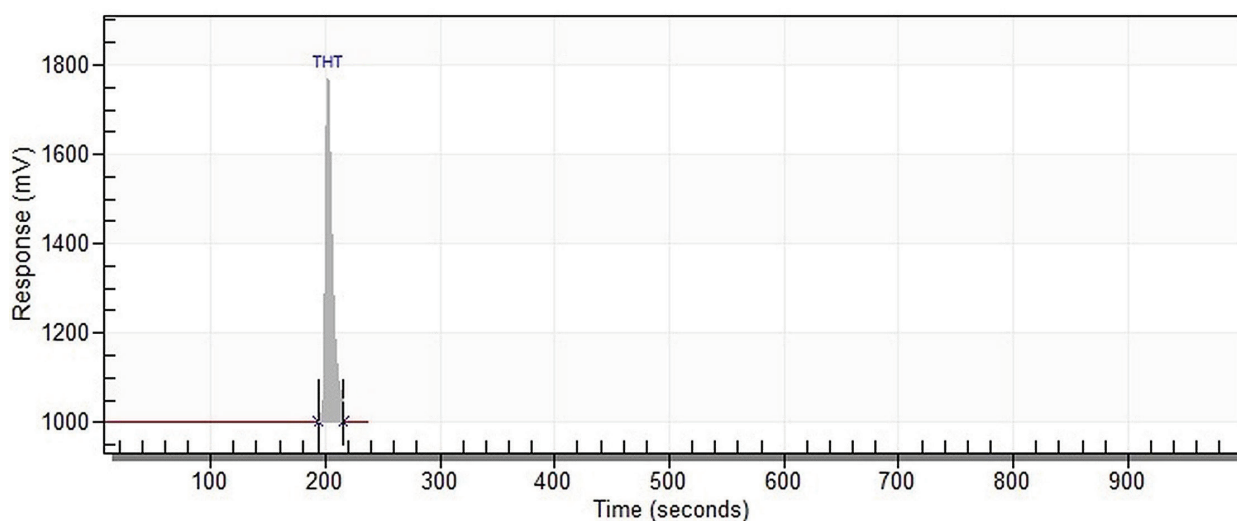


Figure 2 : Chromatogram showing a standard gas containing 9.25ppm THT

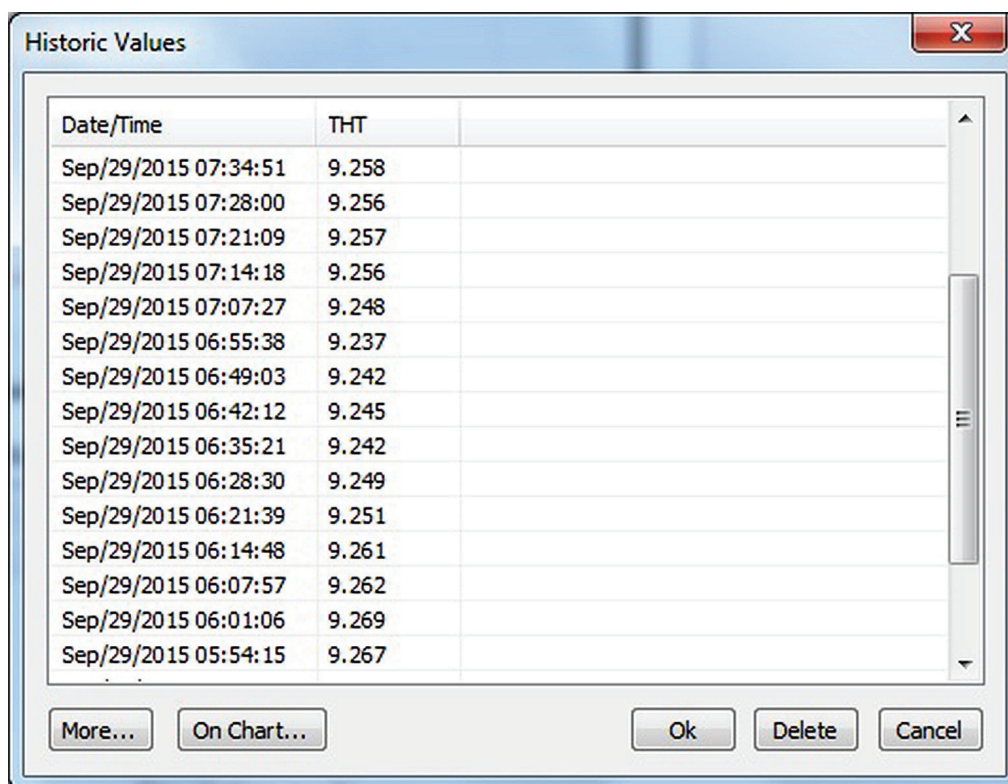


Based on noise to ratio, LDL is calculated as follow:

COMPONENT	CONCENTRATION	PEAK HEIGHT	NOISE	LDL (3X NOISE)
THT	9.257 ppm	1780 mV	2 mv	0.031 ppm

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

Figure 3 : Historic values chart showing repeatability results better than 1% of reading



Date/Time	THT
Sep/29/2015 07:34:51	9.258
Sep/29/2015 07:28:00	9.256
Sep/29/2015 07:21:09	9.257
Sep/29/2015 07:14:18	9.256
Sep/29/2015 07:07:27	9.248
Sep/29/2015 06:55:38	9.237
Sep/29/2015 06:49:03	9.242
Sep/29/2015 06:42:12	9.245
Sep/29/2015 06:35:21	9.242
Sep/29/2015 06:28:30	9.249
Sep/29/2015 06:21:39	9.251
Sep/29/2015 06:14:48	9.261
Sep/29/2015 06:07:57	9.262
Sep/29/2015 06:01:06	9.269
Sep/29/2015 05:54:15	9.267

CONCLUSION:

Using a solution combining the PlasmaDetek2 and MultiDetek2 is the best way to achieve sensitivity, robustness and speed in an industrial compact system. This is exactly what is required from the natural gas industry.



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APPLICATION NOTE

LD16-01



Air analysis using the MultiDetek2 and PlasmaDetek2



▲ MultiDetek2

▲ PlasmaDetek2

LDETEK SOLUTION:

The air analysis for environmental applications is more and more required in different regions of the world. In this application note, the trace analysis of acids, sulfurs and nitrous oxide has been combined in one single compact instrument using one detection technology based on plasma emission (PlasmaDetek2). The MultiDetek2 compact GC has been configured with parallel channels to achieve the measurement at low ppb level for the different impurities. The sample collection can be performed with micro pump for ambient pressure application or bags, with a proportional micro valve for positive pressure application to control flow rate or with our auto injector headspace for vials. The MultiDetek2 was built using heated zones to avoid cold points between the columns and the plasma detector. The detector, valves, fittings and tubing are made of coated stainless steel to avoid surface adsorption. This ensures good sensitivity and repeatability measuring impurities.

RESULTS:

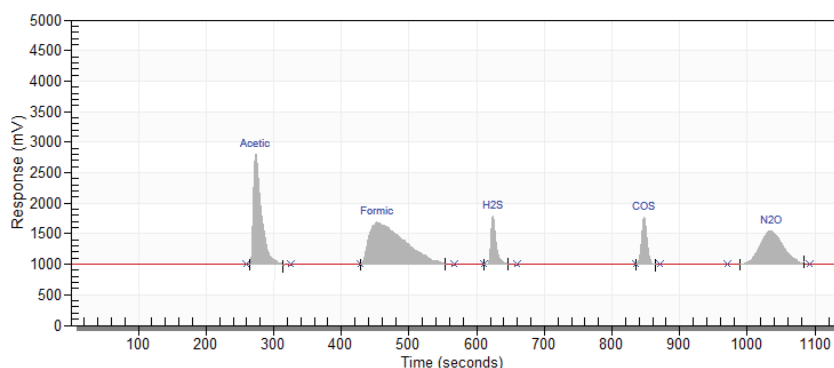
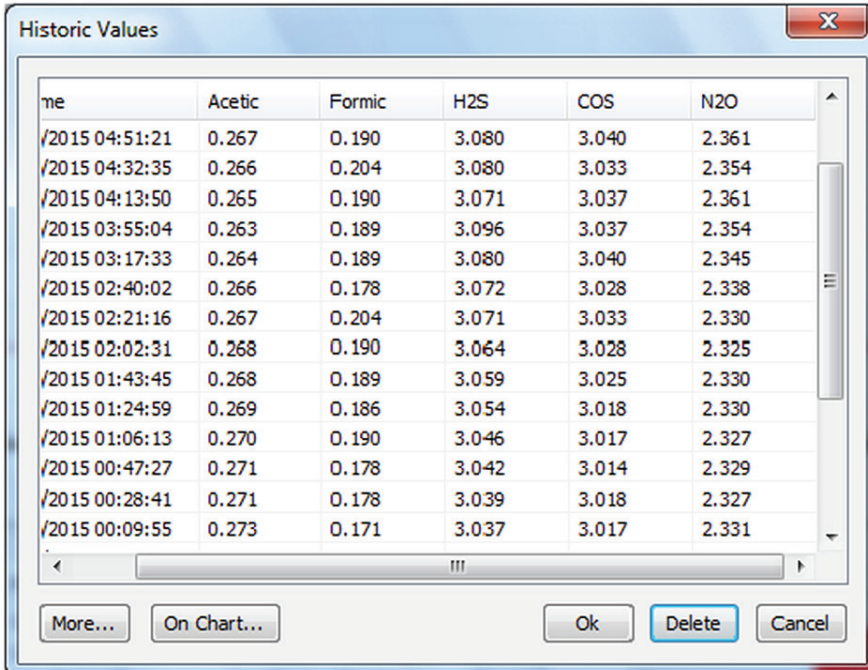


Figure 1:
Chromatogram of trace
impurities in balance air

COMPONENT	CONCENTRATION	PEAK HEIGHT	NOISE	LDL (3X NOISE)
Acetic acid	0.27 ppm	2850 mV	36 mV	0.010 ppm
Formic acid	0.20 ppm	1785 mV	46 mV	0.015 ppm
H2S	3.00 ppm	1860 mV	1.6 mV	0.008 ppm
COS	3.00 ppm	1870 mV	1.9 mV	0.009 ppm
N2O	2.30 ppm	1595 mV	0.4 mV	0.001 ppm

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

Figure 2 : LDL based on 3 times noise ratio ▲



Time	Acetic	Formic	H2S	COS	N2O
/2015 04:51:21	0.267	0.190	3.080	3.040	2.361
/2015 04:32:35	0.266	0.204	3.080	3.033	2.354
/2015 04:13:50	0.265	0.190	3.071	3.037	2.361
/2015 03:55:04	0.263	0.189	3.096	3.037	2.354
/2015 03:17:33	0.264	0.189	3.080	3.040	2.345
/2015 02:40:02	0.266	0.178	3.072	3.028	2.338
/2015 02:21:16	0.267	0.204	3.071	3.033	2.330
/2015 02:02:31	0.268	0.190	3.064	3.028	2.325
/2015 01:43:45	0.268	0.189	3.059	3.025	2.330
/2015 01:24:59	0.269	0.186	3.054	3.018	2.330
/2015 01:06:13	0.270	0.190	3.046	3.017	2.327
/2015 00:47:27	0.271	0.178	3.042	3.014	2.329
/2015 00:28:41	0.271	0.178	3.039	3.018	2.327
/2015 00:09:55	0.273	0.171	3.037	3.017	2.331

**Figure 3 :
Repeatability results**

CONCLUSION:

Using a solution combining the PlasmaDetek 2 and the MultiDetek 2 is the best way to achieve sensitivity, robustness and speed in an industrial or laboratory compact system for air analysis.



Where innovation leads to success

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APPLICATION NOTE

LD16-02



Analysis of hydrocarbons, CO₂, N₂O in Oxygen with the MultiDetek2 compact GC system using Nitrogen as carrier gas and the PlasmaDetek-E detection technology



▲ MultiDetek2

▲ PlasmaDetek2

LDetek SOLUTION:

The hydrocarbon analysis for the production of high purity Oxygen on air separation plants is essential for safety of the operations and quality of the product. For a very long time, the flame ionisation detector has been used for detection of trace hydrocarbons in different gas mixtures. This detector has now many different designs all based on carbon ions collection. The FIDs require a mixture of Air and Hydrogen to generate the flame used for ionisation. It is also necessary to have extra safety based on Hydrogen gas handling. All these points result in increase of operating and start-up costs as demonstrated in Figure 1.

LDetek has developed a detection system based on plasma emission detector (PlasmaDetek E) for the analysis of trace hydrocarbons in different gas mixtures. This PED technology offers enhanced sensitivity and selectivity to hydrocarbons using Nitrogen as single discharge/carrier gas. Other emission and ionisation detection technologies are known in the market for the analysis of hydrocarbons using Argon or Helium as discharge/carrier gas. However, these systems don't offer the selectivity and sensitivity to allow running a single injection through a column without having interferences from residual Oxygen. Other techniques offering NMHC detection are also available and are sufficient in certain cases. Nevertheless, the NMHC detection doesn't give the ability to focus on the measurement of critical impurities like low concentration acetylene in Oxygen production. Using the MultiDetek2 combined with the PlasmaDetek E with Nitrogen carrier gas, the system can be customized for the range and LDL that are specifically required for each impurity. This way, it is possible to configure the system according to each air separation plant specification. As shown in figures 2 and 3, low detection is possible using Nitrogen as carrier gas with PED technology. No need of complex valve and column chromatography configuration for flushing out the background gases.

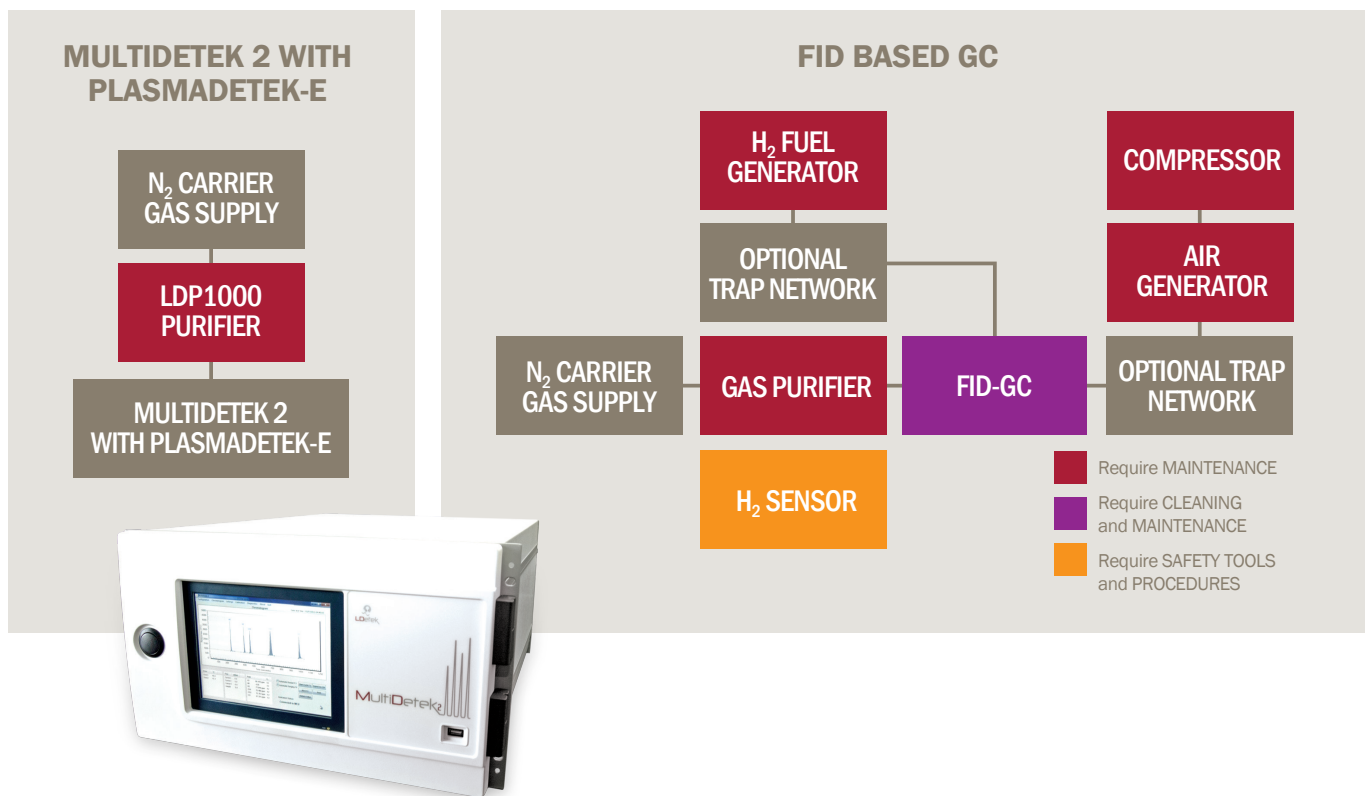


Figure 1 : Comparison between FID based GC and MultiDetek2 compact GC with PlasmaDetek-E

RESULTS:

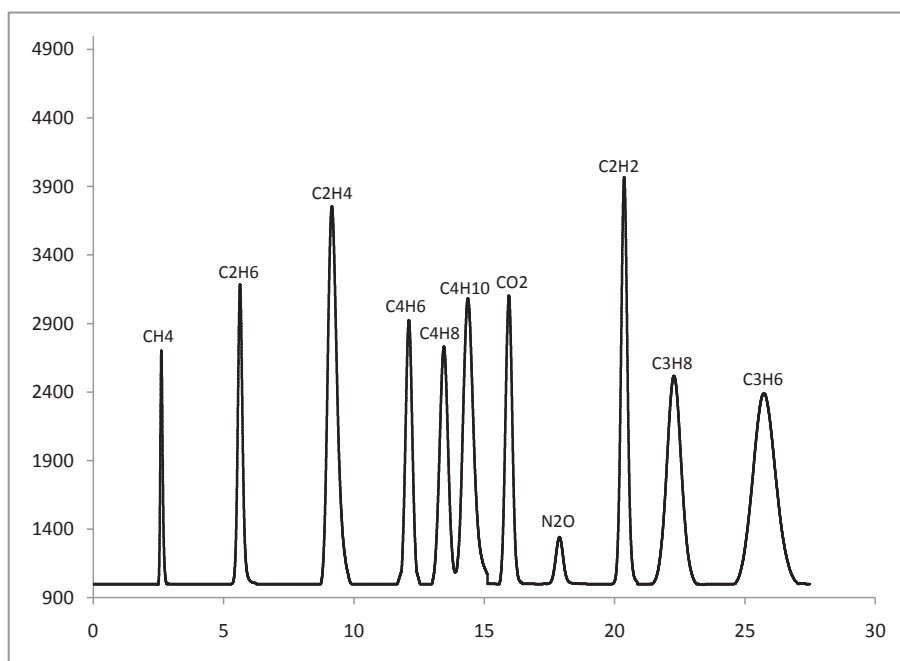


Figure 2 : Chromatogram of trace impurities in balance Oxygen

COMPONENT	CONCENTRATION	PEAK HEIGHT	NOISE	LDL (3X NOISE)
CH4	18.1 ppm	2760 mV	2.1 mV	0.041 ppm
C2H6	8.6 ppm	3308 mV	4.0 mV	0.031 ppm
C2H4	8.2 ppm	3888 mV	4.3 mV	0.027 ppm
C4H6	2.1 ppm	2969 mV	11.0 mV	0.023 ppm
C4H8	1.8 ppm	2798 mV	11.8 mV	0.023 ppm
C4H10	2.1 ppm	3190 mV	11.5 mV	0.023 ppm
CO2	4.1 ppm	3199 mV	11.0 mV	0.042 ppm
N2O	0.8 ppm	1401 mV	3.0 mV	0.005 ppm
C2H2	1.1 ppm	4098 mV	10.5 mV	0.007 ppm
C3H8	8.9 ppm	2559 mV	4.1 mV	0.043 ppm
C3H6	8.7 ppm	2501 mV	3.9 mV	0.041 ppm

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

Figure 3 : LDL based on 3 times noise ratio

CONCLUSION:

With Nitrogen gas used as carrier, the MultiDetek2 with Plasmadetek-E becomes the ideal gas measuring instrument for trace detection of hydrocarbons on the air separation plants for high purity Oxygen monitoring. Keeping the start-up and operating costs low and offering enhanced hydrocarbon detection, LDetek is proud to offer its robust and easy to operate system.



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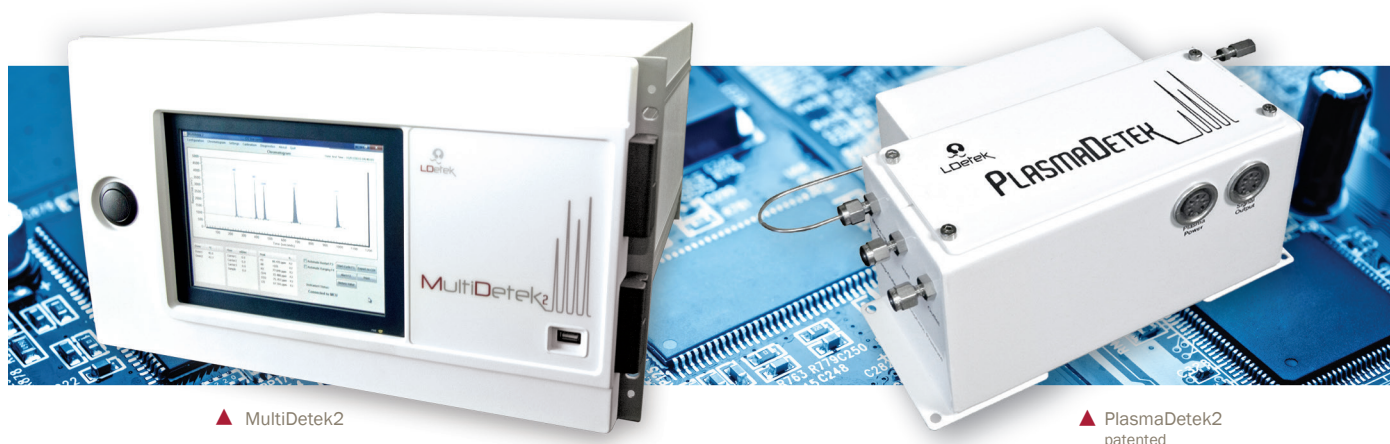
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APPLICATION NOTE

LD16-03



Measurement of part per billion H₂-NMHC-CH₄-N₂-CO₂-CO for semiconductor gases



It is well known in the semiconductor industry that measuring part per billion of permanent gases in ultra high purity gases as Helium, Argon, Oxygen, Nitrogen and Hydrogen is required. Such measurement ensures quality of the product.

LDETEK SOLUTION:

Using the PlasmaDetek2 (PED) and the MultiDetek2 (GC), analyses of part per billion below 1ppb level become feasible all in one chassis. The analyses of the impurities H₂-Ar-N₂-CH₄-CO-CO₂-NMHC at concentration going down to single-digit ppb can be performed in multiple gas backgrounds. This application note will show the results obtained with a MultiDetek2 GC system having multiple configurations.

The MultiDetek2 system detection technology is based on the enhanced plasma emission detector (PlasmaDetek2). The specific configuration of the plasma detector that was used, allows a selective and sensitive detection of the desired impurities and blocks the undesired interference gases. Last years long-term work on the new patented plasma technology used for low ppb detection gives the ability to detect single-digit ppb down to 0.100ppb. It offers the capacity of measuring the complete gas matrix that appears on chart 1, all in one compact industrial GC chassis without the use of any traps as commonly installed by other GC manufacturers.

This document demonstrates the performances of the system by offering chromatograms, charts and graphs all obtained at low ppb concentration to show the real peak shapes and results. For more details about trace ppb Ar-N₂ as impurity, please refer to the application note LD15-02 that gives additional information.

BACKGROUND GASES

IMPURITIES

CONFIGURATION NAME	RANGE	H2 (LDL)	NMHC (LDL)	CH4 (LDL)	N2 (LDL)	CO2 (LDL)	CO (LDL)
Helium	0-500ppb	0.350ppb	0.400ppb	0.300ppb	0.100ppb	0.300pb	0.300ppb
Argon	0-500ppb	0.350ppb	0.400ppb	0.300ppb	0.100ppb	0.300pb	0.300ppb
Oxygen	0-500ppb	0.350ppb	0.550ppb	0.400ppb	0.200ppb	0.400pb	0.400ppb
Hydrogen	0-500ppb	x	0.400ppb	0.400ppb	0.200ppb	0.400pb	0.400ppb
Nitrogen	0-500ppb	0.350ppb	0.400ppb	0.400ppb	x	0.400pb	0.400ppb

Chart 1: MultiDetek2 multiple configuration capabilities

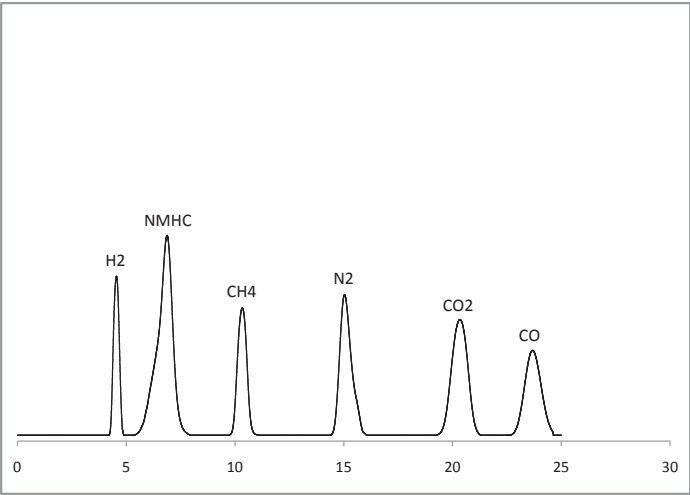
RESULTS:

The results have been demonstrated using the most complicated configuration, which is Oxygen purity analyses. The chromatograms and results have been obtained by measuring different concentrations between 0ppb and 150ppb for the different impurities. It shows and defines the stability, the accuracy, the LDL and the linearity of such system.

ACCURACY AND STABILITY:

The accuracy is demonstrated using 3 different concentrations in the range of 0-500ppb for which ten consecutive cycles have been performed. The delta of the minimum and maximum concentrations measured for the ten consecutive cycles is calculated for each impurity. The accuracy is obtained by dividing the delta on the average results. The accuracy results must be within +/- 0.250ppb or +/-10% of the measured value.

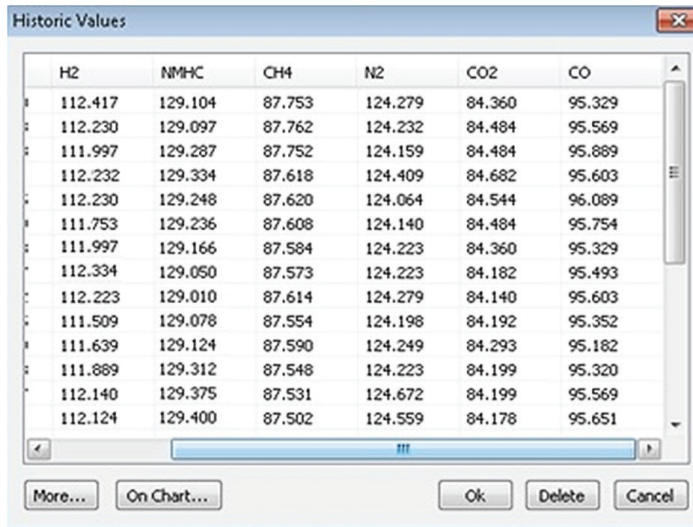
The results indicated on the chromatograms 1, 2, 3 combined with the charts 2, 3, 4 and the figures 1, 2, 3 show well how to interpret the results.



Chromatogram 1:
One chromatogram example
of low ppb trace impurities
used for accuracy and
stability calculation.
Sample gas concentration
appears in chart2.

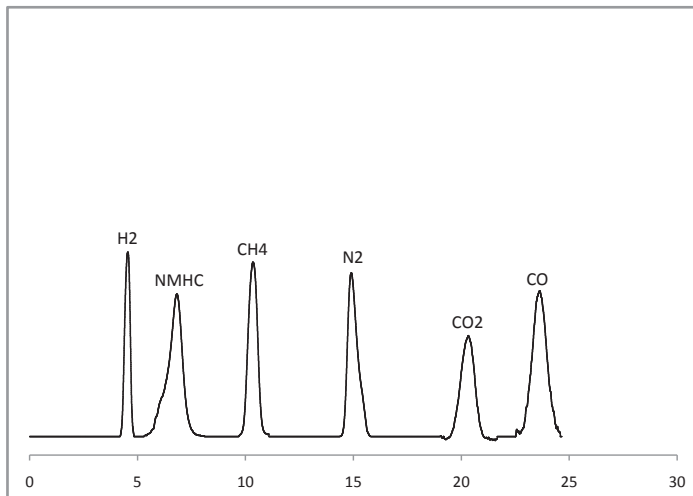
IMPURITIES	MD2 RESULTS PPB (MINIMUM)	MD2 RESULTS PPB (MAXIMUM)	MD2 RESULTS PPB (AVERAGE)	MD2 RESULTS PPB (DELTA MAX-MIN)	ACCURACY (%)
Hydrogen	111.509	112.417	111.963	0.908	+/- 0.4
Non methane hydrocarbons	129.010	129.400	129.205	0.390	+/- 0.15
Methane	87.502	87.762	87.632	0.260	+/- 0.15
Nitrogen	124.064	124.672	124.368	0.608	+/- 0.25
Carbon dioxide	84.140	84.682	84.411	0.542	+/- 0.32
Carbon monoxide	95.182	96.089	95.635	0.907	+/- 0.47

Chart 2: Accuracy & stability (Results are dependent on the system conditions and can vary)



	H2	NMHC	CH4	N2	CO2	CO
1	112.417	129.104	87.753	124.279	84.360	95.329
2	112.230	129.097	87.762	124.232	84.484	95.569
3	111.997	129.287	87.752	124.159	84.484	95.889
4	112.232	129.334	87.618	124.409	84.682	95.603
5	112.230	129.248	87.620	124.064	84.544	96.089
6	111.753	129.236	87.608	124.140	84.484	95.754
7	111.997	129.166	87.584	124.223	84.360	95.329
8	112.334	129.050	87.573	124.223	84.182	95.493
9	112.223	129.010	87.614	124.279	84.140	95.603
10	111.509	129.078	87.554	124.198	84.192	95.352
11	111.639	129.124	87.590	124.249	84.293	95.182
12	111.889	129.312	87.548	124.223	84.199	95.320
13	112.140	129.375	87.531	124.672	84.199	95.569
14	112.124	129.400	87.502	124.559	84.178	95.651

Figure 1 : Results of ten consecutive cycles used for accuracy and stability calculation



Chromatogram 2: One chromatogram example of low ppb trace impurities used for accuracy and stability calculation. Sample gas concentration appears in chart3.

IMPURITIES	MD2 RESULTS PPB (MINIMUM)	MD2 RESULTS PPB (MAXIMUM)	MD2 RESULTS PPB (AVERAGE)	MD2 RESULTS PPB (DELTA MAX-MIN)	ACCURACY (%)
Hydrogen	37.726	38.686	38.206	0.960	+/- 1.25
Non methane hydrocarbons	24.927	25.526	25.226	0.599	+/- 1.19
Methane	36.472	37.150	36.811	0.678	+/- 0.92
Nitrogen	36.728	37.100	36.914	0.372	+/- 0.50
Carbon dioxide	26.103	26.729	26.416	0.626	+/- 1.18
Carbon monoxide	35.845	36.943	36.394	1.098	+/- 1.50

Chart 3: Accuracy & stability (Results are dependent on the system conditions and can vary)

Historic Values

	H2	NMHC	CH4	N2	CO2	CO
38.686	25.526	37.149	36.728	26.477	36.773	
38.562	25.358	37.031	36.821	26.544	36.821	
38.526	25.216	37.150	36.658	26.456	36.730	
38.531	25.280	37.107	36.730	26.103	36.651	
38.646	25.328	37.081	36.845	26.244	36.556	
38.187	25.092	36.990	36.992	26.434	36.773	
38.659	25.150	36.958	36.996	26.283	35.951	
38.642	25.045	36.773	36.920	26.578	35.874	
38.415	24.927	36.651	36.939	26.729	36.587	
37.726	24.992	36.556	36.869	26.662	36.839	
38.173	25.076	36.504	36.951	26.453	36.943	
38.291	25.135	36.501	36.917	26.529	36.032	
37.833	25.198	36.472	37.083	26.246	35.845	
38.321	25.276	36.594	37.100	26.171	35.917	

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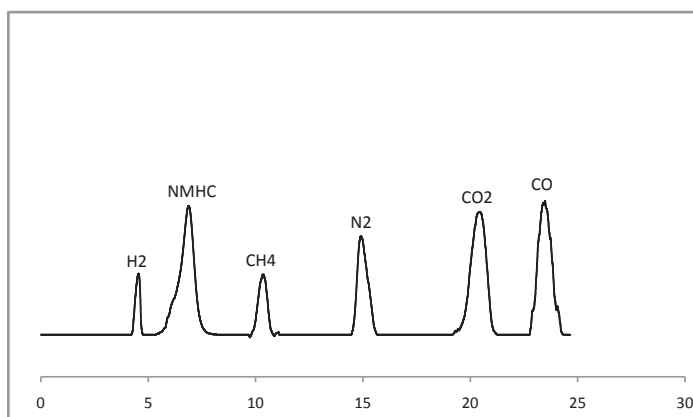
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Figure 2 :
Results of ten consecutive cycles
used for accuracy and stability calculation



Chromatogram 3: One chromatogram example of low ppb trace impurities used for accuracy and stability calculation. Sample gas concentration appears in chart4.

IMPURITIES	MD2 RESULTS PPB (MINIMUM)	MD2 RESULTS PPB (MAXIMUM)	MD2 RESULTS PPB (AVERAGE)	MD2 RESULTS PPB (DELTA MAX-MIN)	ACCURACY (%)
Hydrogen	7.079	8.051	7.565	0.972	+/- 6.42
Non methane hydrocarbons	8.985	9.235	9.110	0.250	+/- 1.37
Methane	5.721	6.517	6.119	0.796	+/- 6.50
Nitrogen	6.895	7.703	7.299	0.808	+/- 5.53
Carbon dioxide	5.648	6.302	5.975	0.654	+/- 5.47
Carbon monoxide	3.708	4.528	4.118	0.820	+/- 9.95

Chart 4: Accuracy & stability (Results are dependent on the system conditions and can vary)

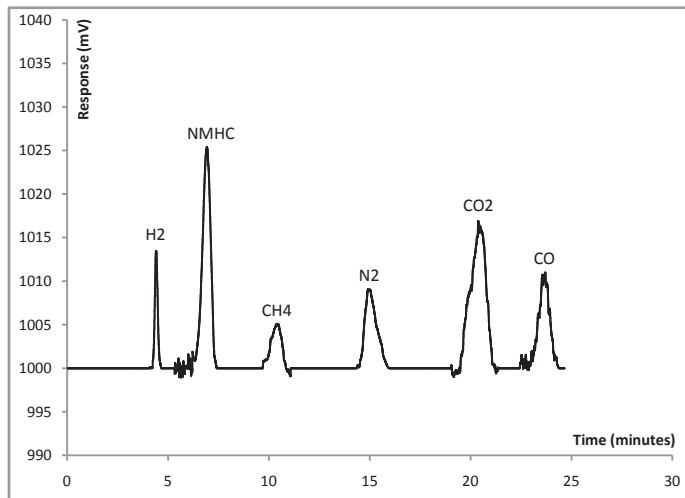
Historic Values						
	H2	NMHC	CH4	N2	CO2	CO
3	7.469	9.170	5.721	7.547	5.747	4.215
2	7.703	9.171	5.825	7.568	5.694	4.314
7	8.051	9.154	6.172	7.549	5.754	4.528
1	7.783	9.124	6.169	7.703	5.932	4.463
5	8.012	9.077	6.257	7.700	6.104	4.386
9	7.789	9.020	6.266	7.687	6.302	4.388
2	7.606	8.985	5.823	7.682	6.176	4.275
5	7.343	9.026	6.311	7.642	6.062	4.056
1	7.403	9.071	6.309	7.522	5.862	4.068
5	7.162	9.140	6.334	7.426	5.848	4.077
9	7.123	9.229	6.517	7.547	5.648	4.143
8	7.079	9.235	6.234	7.091	5.660	3.708
1	7.703	9.164	6.344	7.011	5.709	4.234
2	7.364	9.098	6.373	6.895	6.200	4.364

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Figure 3 :
Results of ten consecutive cycles used for
accuracy and stability calculation

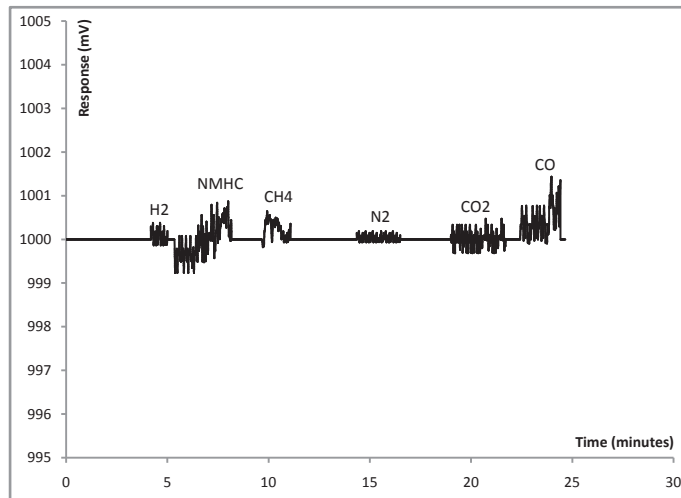
LOWER DETECTION LIMIT (LDL):

The limit of detection is identified by running an analysis below 5ppb of each impurity to identify the peak response height. Then, noise baseline identification is performed by running a cycle without valve actuation and measuring the noise level during the integration time of each impurity. The limit of detection (LDL) for each impurity is the relation between the peak response at a value below 5ppb and three times the noise level identified with baseline analysis. The results indicated on the chromatograms 4 & 5 combined with the chart 5 show well the performance of the system.



Chromatogram 4:

One chromatogram example of low ppb trace impurities used for LDL calculation. Sample gas concentration appears in chart5.



Chromatogram 5:

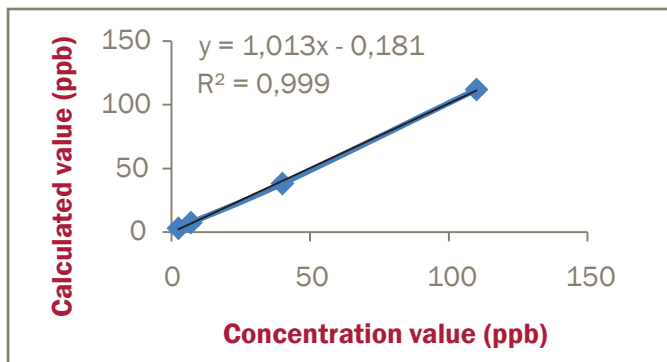
Baseline noise analysis used for LDL calculation

IMPURITIES	SAMPLE CONCENTRATION (PPB)	RESPONSE (MV)	NOISE (MV)	3X NOISE (MV)	LDL (PPB) (3X NOISE LEVEL)
Hydrogen	3.1	13.49	0.48	1.44	0.331
Non methane hydrocarbons	3.2	25.34	1.47	4.41	0.556
Methane	0.9	5.01	0.74	2.22	0.398
Nitrogen	2.1	9.06	0.23	0.69	0.160
Carbon dioxide	2.3	16.93	0.76	2.28	0.309
Carbon monoxide	1.0	11	1.42	4.26	0.387

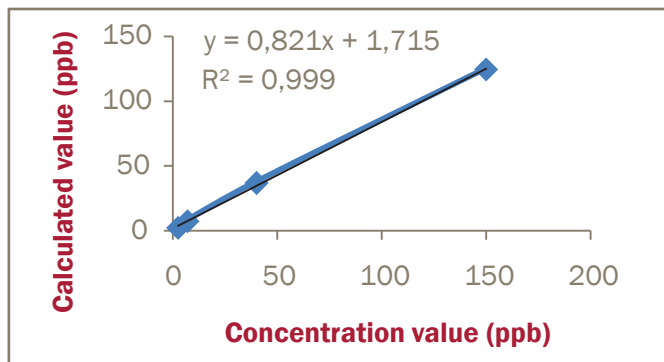
Chart 5: LDL calculation (results are dependent on the system conditions and can vary)

LINEARITY :

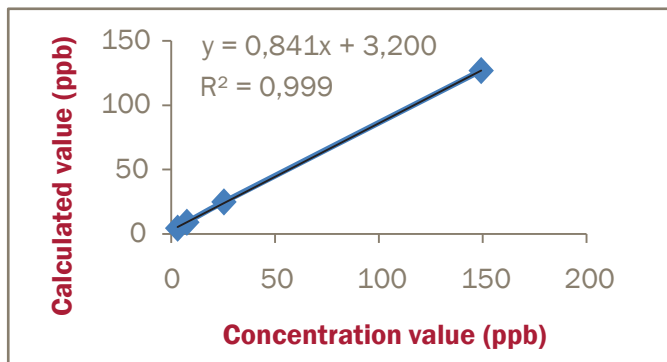
The linearity is calculated by running a minimum of 10 cycles at four different concentrations between 0 and 500ppb. The average of the results of the 10 cycles is used to generate the calculated values which are compared to a linear trend in relation to the known concentration values. The graphs 1-2-3-4-5 and 6 show well the good linearity results for each impurity.



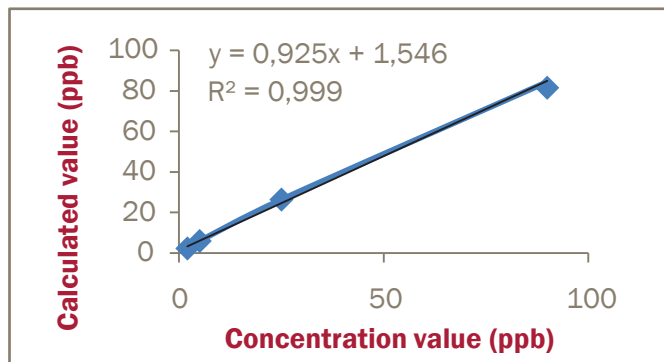
Graph 1: H₂ peak linearity



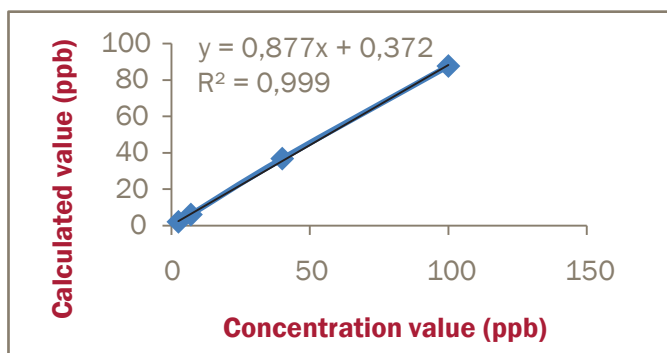
Graph 4: N₂ peak linearity



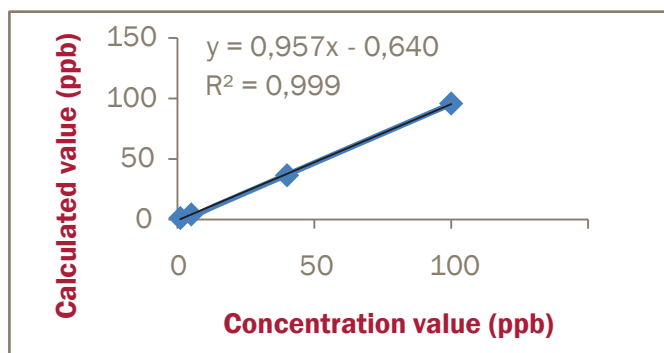
Graph 2: NMHC peak linearity



Graph 5: CO₂ peak linearity



Graph 3: CH₄ peak linearity



Graph 6: CO peak linearity

CONCLUSION:

Combined with the patented PlasmaDetek2, the compact, industrial and rack mount GC MultiDetek2 is a great instrument when analysis of low ppb is required. This compact system is able to manage multiple background gases in a single 6U chassis. No need of extra trap system. With its industrial design, the MultiDetek2 can manage all standard industrial communication protocols and remotely control the streams with communication with high purity LDGSS stream selector. Its remote access also gives the ability to easily support the instrument from distance. Many more features are available in this system what makes it ideal for the semiconductor market..



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APPLICATION NOTE

LD16-13



Measurement of Greenhouse gases GHG



Energy (fossil fuel) and agriculture required for human activities on Earth produce Greenhouse gases (GHG) such as Carbon Dioxide (CO₂), Methane (CH₄), Nitrous Oxide (N₂O) and Fluorinated gases mainly Sulfur Hexafluoride (SF₆). These gases tend to absorb infrared radiation emitted by the Earth surface resulting to reduce the atmospheric heat loss into space and keeping Earth warmer.

The source of every GHG type is associated to different sectors resulting of human activities:

CO₂ → *Fossil fuel, industrial processes, forestry, land use for agriculture*

CH₄ → *Agriculture, waste management, energy use, biomass burning*

N₂O → *Agriculture (such as fertilizer use) and biomass burning*

F-gases (SF₆) → *Industrial processes and refrigeration*

For this reason, the Kyoto Protocol was established in 1997 by the industrialized countries with an objective to measure, control and decrease the GHG in order to reduce the continuous global warming.

Figures 1, 2 and 3 show some data that demonstrate the impact of human activities to increase the Greenhouse gases level worldwide.

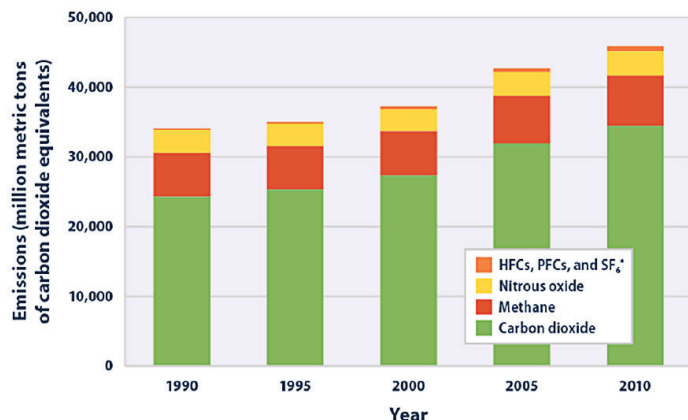


Figure 1:
Global Greenhouse gas emissions by gas type,
1990-2010

(Data sources WRI 2014, FAO 2014) ¹

This figure shows worldwide emissions of carbon dioxide, methane, nitrous oxide and several fluorinated gases from 1990 to 2010. For consistency, emissions are expressed in million metric tons of carbon dioxide equivalents. These totals include emissions and sinks due to land-use change and forestry.

*HFCs are hydro fluorocarbons, PFCs are per fluorocarbons and SF₆ is hexafluoride

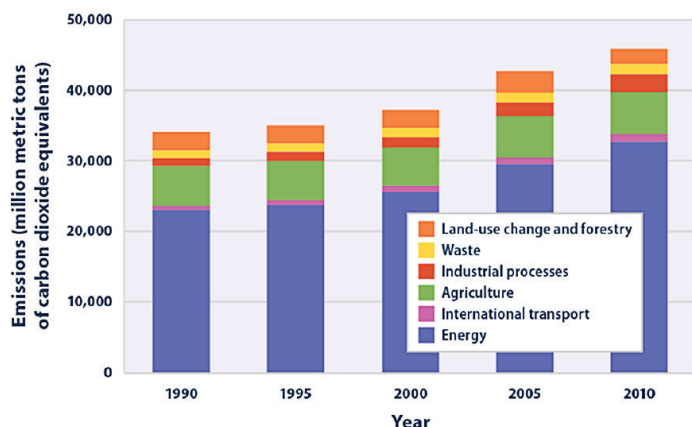


Figure 2:
Global Greenhouse gas emissions by sector,
1990-2010

(Data sources WRI 2014, FAO 2014) ¹

This figure shows worldwide gas emissions by sector from 1990 to 2010.

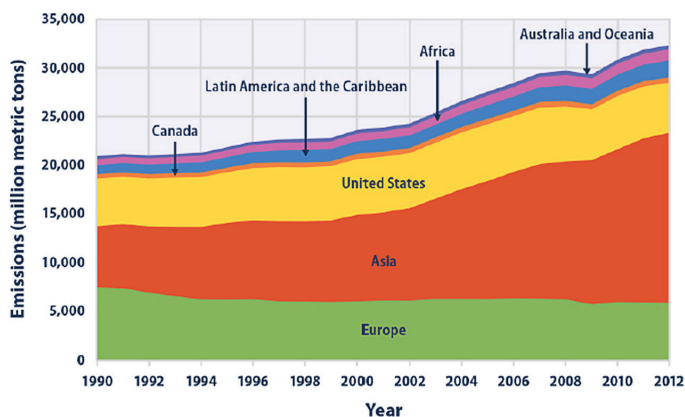


Figure 3:
Global Greenhouse gas emissions by regions,
1990-2012

(Data sources WRI 2015) ¹

This figure shows carbon dioxide emissions from 1990 to 2012 for different regions of the world.

¹ Data and analysis come from the World Resources Institute's Climate Analysis Indicators Tool (CAIT), which compiles data from peer reviewed and internationally recognized greenhouse gas inventories developed by EPA and other government agencies worldwide.

The well known technique to measure and quantify the GHG is Gas Chromatography. It can determine the rate of emission or absorption. These rates are mainly measured using samples of soil, rice, maize or wheat from different areas. The measurement of ambient air is also used. The worldwide GHG can then be better measured and controlled to facilitate the proper actions to be taken to reduce the global warming.

ALTERNATIVE SOLUTION USING GAS CHROMATOGRAPH

The use of multiple detectors in a gas chromatography system is generally necessary to cover the greenhouse application.

A FID detector and a methanizer system for detection of trace impurities of Methane (CH₄) and Carbon Dioxide (CO₂). Such detector requires Fuel (H₂), Air and the carrier gas source to make it works.

An ECD detector to measure traces Nitrous Oxide (N₂O) and Sulfur Hexafluoride (SF₆). This type of detector is a source of radioactivity and results in complication for storage, handling and transport.

A TCD is also required for measuring high concentration Carbon Dioxide (CO₂) when necessary.

It is then a solution combining multiple types of detection technology which makes the system more complex to operate and increases the operating cost.

OUR SOLUTION

Using the PlasmaDetek2 (PED) plasma detector and the MultiDetek2 compact gas chromatograph, the analysis of the most critical trace impurities in air can be achieved in one unit with a single detection technology (PED).

By default, the configuration has 2 channels to cover the analysis of Methane, Carbon Dioxide, Nitrous Oxide and Sulfur Hexafluoride. If additional options for the analysis of other impurities like fluorinated gases (F-gases) are required, then an additional channel still using a Plasma Emission Detector can be added in the same instrument.

Channel#1: This channel measures 0-1000ppm Methane (CH₄) and 0-5000ppm Carbon Dioxide (CO₂) using the PlasmaDetek2 (PED). Other measuring ranges can be configured. The system is protected against moisture using a pre column in back flush to vent configuration.

Channel#2: 0-10ppm Sulfur Hexafluoride (SF₆) and Nitrous Oxide (N₂O) still uses the PlasmaDetek2 (PED). Other ranges are also possible. A pre column to protect the system against moisture is also used in this channel.

Channel#3 (optional): Analysis of other fluorinated gases (F-gases) that would still be using the PlasmaDetek2 (PED) as detector.

The sample preparation is managed by the HTA Headspace auto sampler system mounted on the MultiDetek2 GC. The auto sampler tray configuration can be for 14 or 42 vials having size range of 10ml or 20ml depending of the application requirements. The oven can accept 1 or 6 vials for heating and shaking in preparation of analysis. The auto sampler offers syringe auto cleaning and a purge routine in between each sample collection. The syringe volume can be 2.5ml or 5ml depending of the application.

The Headspace can be controlled from its keypad, touch screen interface or from the software environment installed on an external PC or in the integrated PC of the MultiDetek2.

The data management is done with the MultiDetek2 interface. The unit can be control locally from the MultiDetek2 touch screen interface or remotely using LAN connectivity.

RESULTS

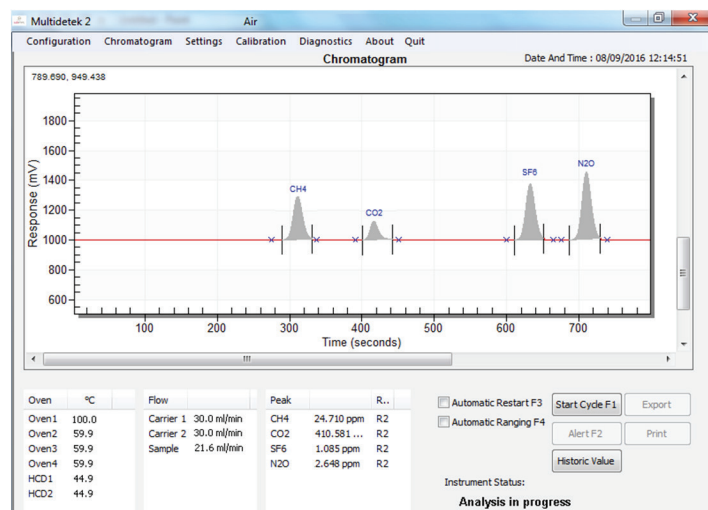


Figure 1: chromatogram of trace impurities in balance air coming from a certified gas bottle.

For this application, a measuring range of 0-1000ppm was required for the CH₄ with a LDL of 200ppb.

A measuring range of 0-5000ppm was required for the CO₂ with a LDL of 500ppb.

A measuring range of 10ppm was required for the SF₆ and N₂O with a LDL of 20ppb.

The figure 2 shows a table where the LDL calculation was obtained with a noise/response ratio. It shows the system can achieve better specifications than what was required for the application. Lower detection limits can be obtained with the same system just by changing the injected sampling loop volume. An extended range with lower detection limit capabilities can be obtained with our optional multiple sampling loop injection system.

It becomes possible to measure ppt/ppb/high ppm with the same detector in the same instrument.

COMPONENT	CONCENTRATION	PEAK HEIGHT	NOISE	LDL (3X NOISE)
CH4	24.710 ppm	331 mV	0.4 mV	89.6 ppb
CO2	410.581 ppm	177 mV	0.06 mV	417.5 ppb
SF6	1.085 ppm	389 mV	1.6 mV	13.4 ppb
N2O	2.648 ppm	479 mV	0.9 mV	14.9 ppb

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

Figure 2

CONCLUSION

The combination of the PlasmaDetek2 (PED) detector with the MultiDetek2 compact gas chromatograph and the HTA Headspace auto sampler becomes an interesting reliable and robust solution for the laboratory requirements where Greenhouse (GHG) gases must be measured.

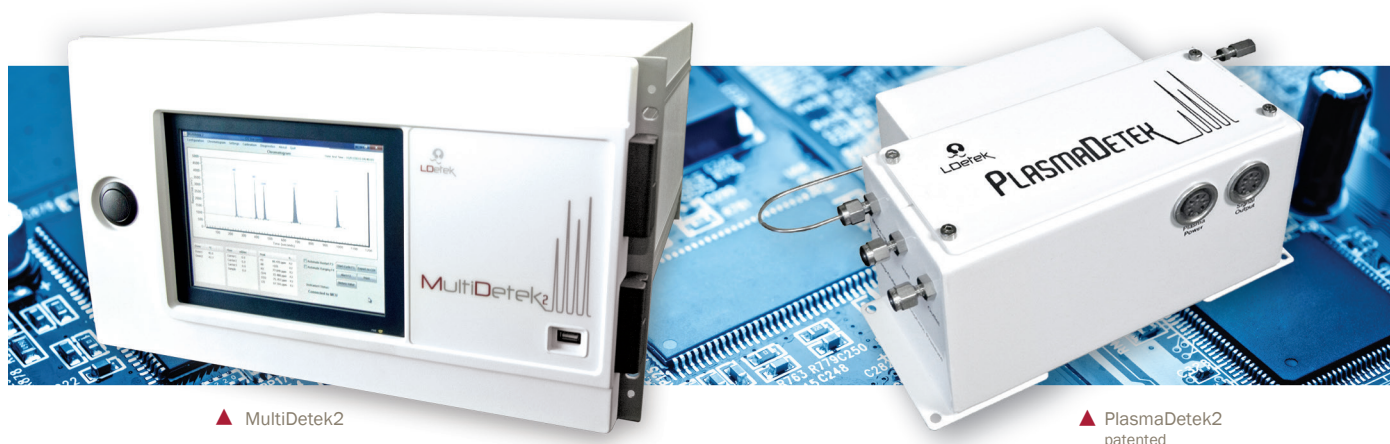


APPLICATION NOTE

LD16-03



Measurement of part per billion H₂-NMHC-CH₄-N₂-CO₂-CO for semiconductor gases



It is well known in the semiconductor industry that measuring part per billion of permanent gases in ultra high purity gases as Helium, Argon, Oxygen, Nitrogen and Hydrogen is required. Such measurement ensures quality of the product.

LDETEK SOLUTION:

Using the PlasmaDetek2 (PED) and the MultiDetek2 (GC), analyses of part per billion below 1ppb level become feasible all in one chassis. The analyses of the impurities H₂-Ar-N₂-CH₄-CO-CO₂-NMHC at concentration going down to single-digit ppb can be performed in multiple gas backgrounds. This application note will show the results obtained with a MultiDetek2 GC system having multiple configurations.

The MultiDetek2 system detection technology is based on the enhanced plasma emission detector (PlasmaDetek2). The specific configuration of the plasma detector that was used, allows a selective and sensitive detection of the desired impurities and blocks the undesired interference gases. Last years long-term work on the new patented plasma technology used for low ppb detection gives the ability to detect single-digit ppb down to 0.100ppb. It offers the capacity of measuring the complete gas matrix that appears on chart 1, all in one compact industrial GC chassis without the use of any traps as commonly installed by other GC manufacturers.

This document demonstrates the performances of the system by offering chromatograms, charts and graphs all obtained at low ppb concentration to show the real peak shapes and results. For more details about trace ppb Ar-N₂ as impurity, please refer to the application note LD15-02 that gives additional information.

BACKGROUND GASES

IMPURITIES

CONFIGURATION NAME	RANGE	H2 (LDL)	NMHC (LDL)	CH4 (LDL)	N2 (LDL)	CO2 (LDL)	CO (LDL)
Helium	0-500ppb	0.350ppb	0.400ppb	0.300ppb	0.100ppb	0.300pb	0.300ppb
Argon	0-500ppb	0.350ppb	0.400ppb	0.300ppb	0.100ppb	0.300pb	0.300ppb
Oxygen	0-500ppb	0.350ppb	0.550ppb	0.400ppb	0.200ppb	0.400pb	0.400ppb
Hydrogen	0-500ppb	x	0.400ppb	0.400ppb	0.200ppb	0.400pb	0.400ppb
Nitrogen	0-500ppb	0.350ppb	0.400ppb	0.400ppb	x	0.400pb	0.400ppb

Chart 1: MultiDetek2 multiple configuration capabilities

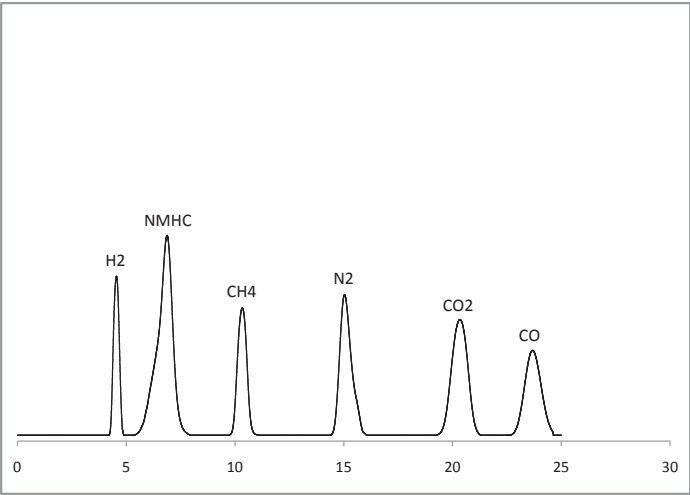
RESULTS:

The results have been demonstrated using the most complicated configuration, which is Oxygen purity analyses. The chromatograms and results have been obtained by measuring different concentrations between 0ppb and 150ppb for the different impurities. It shows and defines the stability, the accuracy, the LDL and the linearity of such system.

ACCURACY AND STABILITY:

The accuracy is demonstrated using 3 different concentrations in the range of 0-500ppb for which ten consecutive cycles have been performed. The delta of the minimum and maximum concentrations measured for the ten consecutive cycles is calculated for each impurity. The accuracy is obtained by dividing the delta on the average results. The accuracy results must be within +/- 0.250ppb or +/-10% of the measured value.

The results indicated on the chromatograms 1, 2, 3 combined with the charts 2, 3, 4 and the figures 1, 2, 3 show well how to interpret the results.



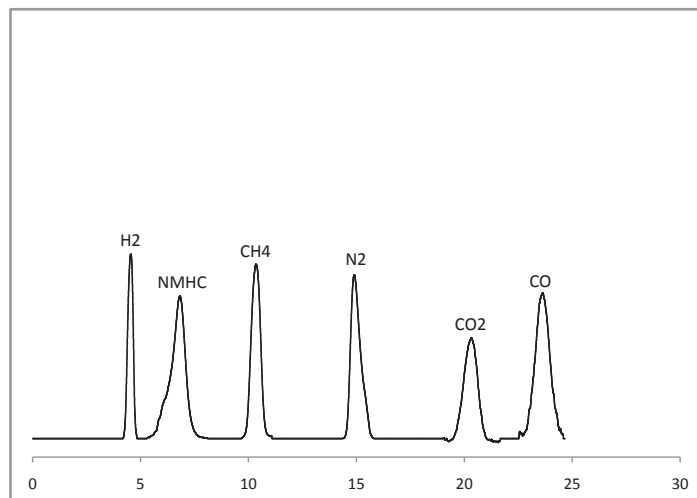
Chromatogram 1:
One chromatogram example
of low ppb trace impurities
used for accuracy and
stability calculation.
Sample gas concentration
appears in chart2.

IMPURITIES	MD2 RESULTS PPB (MINIMUM)	MD2 RESULTS PPB (MAXIMUM)	MD2 RESULTS PPB (AVERAGE)	MD2 RESULTS PPB (DELTA MAX-MIN)	ACCURACY (%)
Hydrogen	111.509	112.417	111.963	0.908	+/- 0.4
Non methane hydrocarbons	129.010	129.400	129.205	0.390	+/- 0.15
Methane	87.502	87.762	87.632	0.260	+/- 0.15
Nitrogen	124.064	124.672	124.368	0.608	+/- 0.25
Carbon dioxide	84.140	84.682	84.411	0.542	+/- 0.32
Carbon monoxide	95.182	96.089	95.635	0.907	+/- 0.47

Chart 2: Accuracy & stability (Results are dependent on the system conditions and can vary)

H2	NMHC	CH4	N2	CO2	CO
112.417	129.104	87.753	124.279	84.360	95.329
112.230	129.097	87.762	124.232	84.484	95.569
111.997	129.287	87.752	124.159	84.484	95.889
112.232	129.334	87.618	124.409	84.682	95.603
112.230	129.248	87.620	124.064	84.544	96.089
111.753	129.236	87.608	124.140	84.484	95.754
111.997	129.166	87.584	124.223	84.360	95.329
112.334	129.050	87.573	124.223	84.182	95.493
112.223	129.010	87.614	124.279	84.140	95.603
111.509	129.078	87.554	124.198	84.192	95.352
111.639	129.124	87.590	124.249	84.293	95.182
111.889	129.312	87.548	124.223	84.199	95.320
112.140	129.375	87.531	124.672	84.199	95.569
112.124	129.400	87.502	124.559	84.178	95.651

Figure 1 : Results of ten consecutive cycles used for accuracy and stability calculation



Chromatogram 2: One chromatogram example of low ppb trace impurities used for accuracy and stability calculation. Sample gas concentration appears in chart3.

IMPURITIES	MD2 RESULTS PPB (MINIMUM)	MD2 RESULTS PPB (MAXIMUM)	MD2 RESULTS PPB (AVERAGE)	MD2 RESULTS PPB (DELTA MAX-MIN)	ACCURACY (%)
Hydrogen	37.726	38.686	38.206	0.960	+/- 1.25
Non methane hydrocarbons	24.927	25.526	25.226	0.599	+/- 1.19
Methane	36.472	37.150	36.811	0.678	+/- 0.92
Nitrogen	36.728	37.100	36.914	0.372	+/- 0.50
Carbon dioxide	26.103	26.729	26.416	0.626	+/- 1.18
Carbon monoxide	35.845	36.943	36.394	1.098	+/- 1.50

Chart 3: Accuracy & stability (Results are dependent on the system conditions and can vary)

Historic Values

	H2	NMHC	CH4	N2	CO2	CO
38.686	25.526	37.149	36.728	26.477	36.773	
38.562	25.358	37.031	36.821	26.544	36.821	
38.526	25.216	37.150	36.658	26.456	36.730	
38.531	25.280	37.107	36.730	26.103	36.651	
38.646	25.328	37.081	36.845	26.244	36.556	
38.187	25.092	36.990	36.992	26.434	36.773	
38.659	25.150	36.958	36.996	26.283	35.951	
38.642	25.045	36.773	36.920	26.578	35.874	
38.415	24.927	36.651	36.939	26.729	36.587	
37.726	24.992	36.556	36.869	26.662	36.839	
38.173	25.076	36.504	36.951	26.453	36.943	
38.291	25.135	36.501	36.917	26.529	36.032	
37.833	25.198	36.472	37.083	26.246	35.845	
38.321	25.276	36.594	37.100	26.171	35.917	

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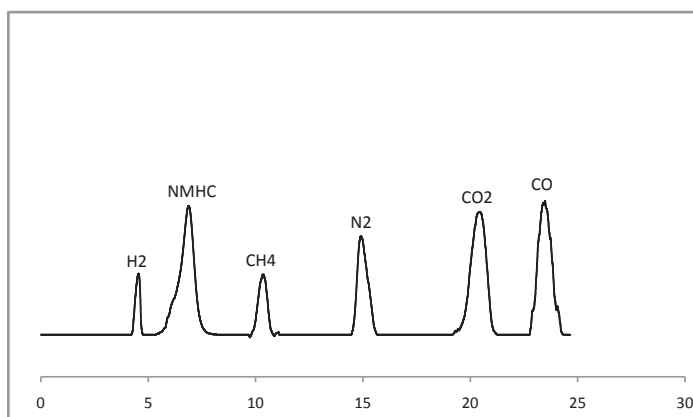
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Figure 2 :
Results of ten consecutive cycles
used for accuracy and stability calculation



Chromatogram 3: One chromatogram example of low ppb trace impurities used for accuracy and stability calculation. Sample gas concentration appears in chart4.

IMPURITIES	MD2 RESULTS PPB (MINIMUM)	MD2 RESULTS PPB (MAXIMUM)	MD2 RESULTS PPB (AVERAGE)	MD2 RESULTS PPB (DELTA MAX-MIN)	ACCURACY (%)
Hydrogen	7.079	8.051	7.565	0.972	+/- 6.42
Non methane hydrocarbons	8.985	9.235	9.110	0.250	+/- 1.37
Methane	5.721	6.517	6.119	0.796	+/- 6.50
Nitrogen	6.895	7.703	7.299	0.808	+/- 5.53
Carbon dioxide	5.648	6.302	5.975	0.654	+/- 5.47
Carbon monoxide	3.708	4.528	4.118	0.820	+/- 9.95

Chart 4: Accuracy & stability (Results are dependent on the system conditions and can vary)

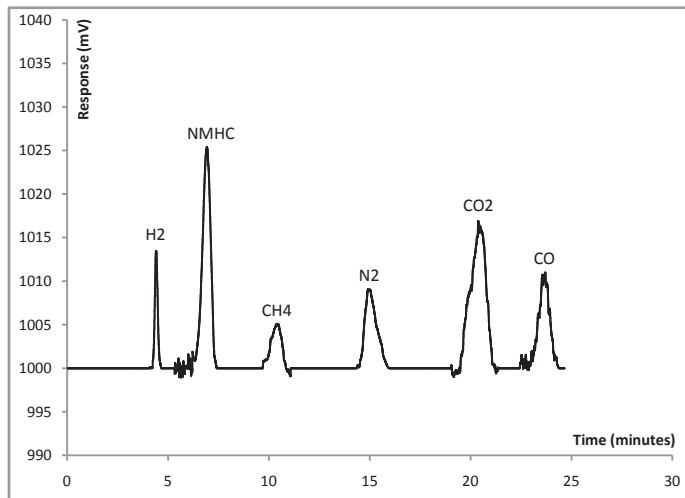
Historic Values						
	H2	NMHC	CH4	N2	CO2	CO
3	7.469	9.170	5.721	7.547	5.747	4.215
2	7.703	9.171	5.825	7.568	5.694	4.314
7	8.051	9.154	6.172	7.549	5.754	4.528
1	7.783	9.124	6.169	7.703	5.932	4.463
5	8.012	9.077	6.257	7.700	6.104	4.386
9	7.789	9.020	6.266	7.687	6.302	4.388
2	7.606	8.985	5.823	7.682	6.176	4.275
5	7.343	9.026	6.311	7.642	6.062	4.056
1	7.403	9.071	6.309	7.522	5.862	4.068
5	7.162	9.140	6.334	7.426	5.848	4.077
9	7.123	9.229	6.517	7.547	5.648	4.143
8	7.079	9.235	6.234	7.091	5.660	3.708
1	7.703	9.164	6.344	7.011	5.709	4.234
2	7.364	9.098	6.373	6.895	6.200	4.364

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Figure 3 :
Results of ten consecutive cycles used for
accuracy and stability calculation

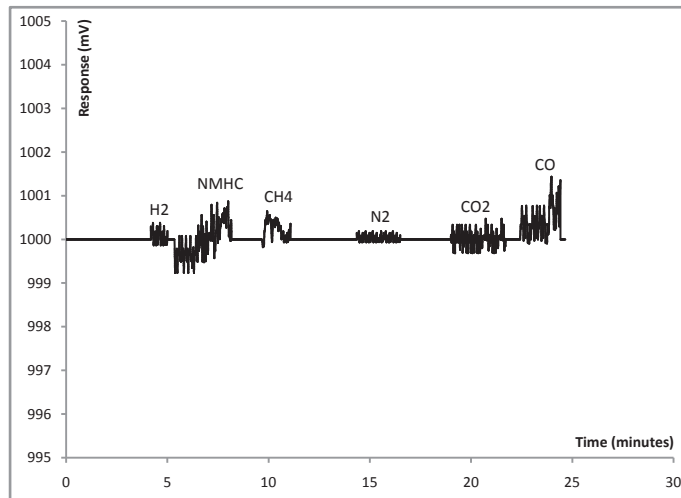
LOWER DETECTION LIMIT (LDL):

The limit of detection is identified by running an analysis below 5ppb of each impurity to identify the peak response height. Then, noise baseline identification is performed by running a cycle without valve actuation and measuring the noise level during the integration time of each impurity. The limit of detection (LDL) for each impurity is the relation between the peak response at a value below 5ppb and three times the noise level identified with baseline analysis. The results indicated on the chromatograms 4 & 5 combined with the chart 5 show well the performance of the system.



Chromatogram 4:

One chromatogram example of low ppb trace impurities used for LDL calculation. Sample gas concentration appears in chart5.



Chromatogram 5:

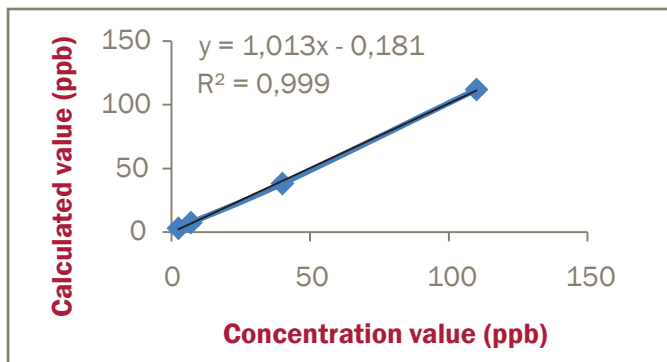
Baseline noise analysis used for LDL calculation

IMPURITIES	SAMPLE CONCENTRATION (PPB)	RESPONSE (MV)	NOISE (MV)	3X NOISE (MV)	LDL (PPB) (3X NOISE LEVEL)
Hydrogen	3.1	13.49	0.48	1.44	0.331
Non methane hydrocarbons	3.2	25.34	1.47	4.41	0.556
Methane	0.9	5.01	0.74	2.22	0.398
Nitrogen	2.1	9.06	0.23	0.69	0.160
Carbon dioxide	2.3	16.93	0.76	2.28	0.309
Carbon monoxide	1.0	11	1.42	4.26	0.387

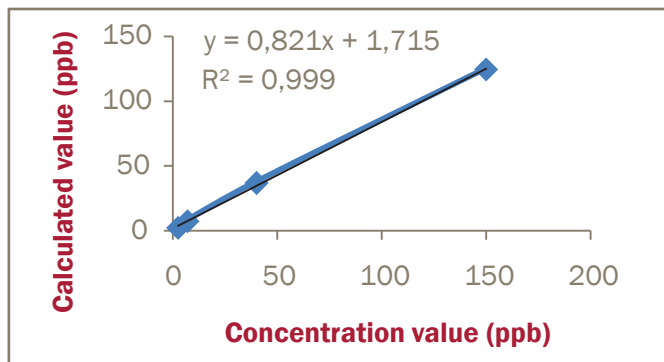
Chart 5: LDL calculation (results are dependent on the system conditions and can vary)

LINEARITY :

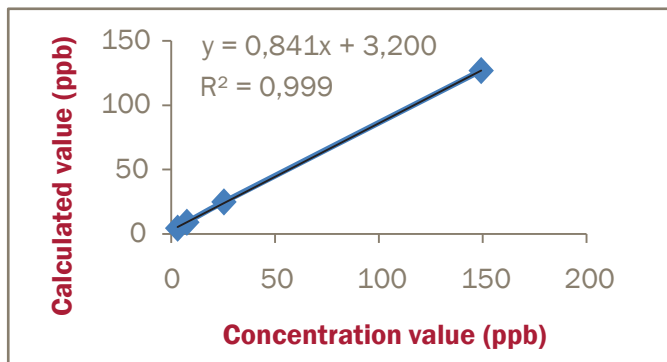
The linearity is calculated by running a minimum of 10 cycles at four different concentrations between 0 and 500ppb. The average of the results of the 10 cycles is used to generate the calculated values which are compared to a linear trend in relation to the known concentration values. The graphs 1-2-3-4-5 and 6 show well the good linearity results for each impurity.



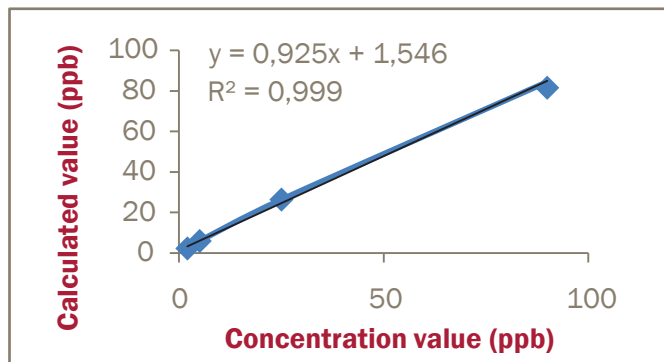
Graph 1: H₂ peak linearity



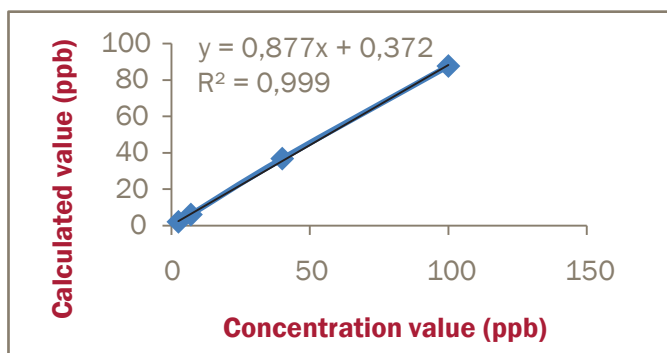
Graph 4: N₂ peak linearity



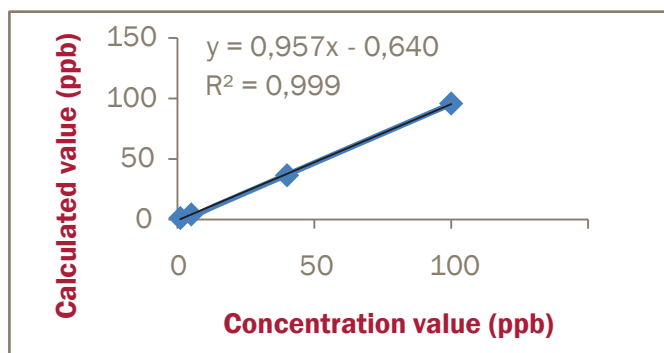
Graph 2: NMHC peak linearity



Graph 5: CO₂ peak linearity



Graph 3: CH₄ peak linearity



Graph 6: CO peak linearity

CONCLUSION:

Combined with the patented PlasmaDetek2, the compact, industrial and rack mount GC MultiDetek2 is a great instrument when analysis of low ppb is required. This compact system is able to manage multiple background gases in a single 6U chassis. No need of extra trap system. With its industrial design, the MultiDetek2 can manage all standard industrial communication protocols and remotely control the streams with communication with high purity LDGSS stream selector. Its remote access also gives the ability to easily support the instrument from distance. Many more features are available in this system what makes it ideal for the semiconductor market..



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APPLICATION NOTE

LD16-04



Sulfur Hexafluoride (SF₆) purity analysis using the MultiDetek 2 and PlasmaDetek 2



SF₆ is used in the electrical industry as a gaseous dielectric medium for high-voltage circuit breakers, switchgear and other electrical equipment. Due to its high electrical insulation properties, it is often used for replacing oil filled circuit breakers. The sulfur hexafluoride is an expensive gas and it also has been identified as the most potent greenhouse gas. A SF₆ purity monitoring instrument is then required in the electrical industry to prevent failures, extend equipment life, reduces equipment cost and increase safety.

LDETEK SOLUTION:

Using the compact MultiDetek2 combined with the robust PlasmaDetek2 detector, this application note shows our solution for measuring impurities in SF₆ to ensure good operations of electrical equipment. The system has been configured with two channels both merging in the PlasmaDetek2 detector.

The first channel is used for measuring O₂-N₂-CO using a porous polymer Q type pre column combined with a Mol Sieve separation column. The pre column is mounted on a 10 port injection/back flush diaphragm valve to flush out the SF₆. The second column allows the separation of O₂-N₂ and CO.

The second channel is used for CF₄ analysis. This channel is also mounted with a 10 port injection/back flush diaphragm valve with two Porapak Q type columns. The pre column is used to flush out the SF₆ and the second column for separation of CF₄ from other impurities. This channel can also allow the analysis of CO₂ and SOF₂.

RESULTS:

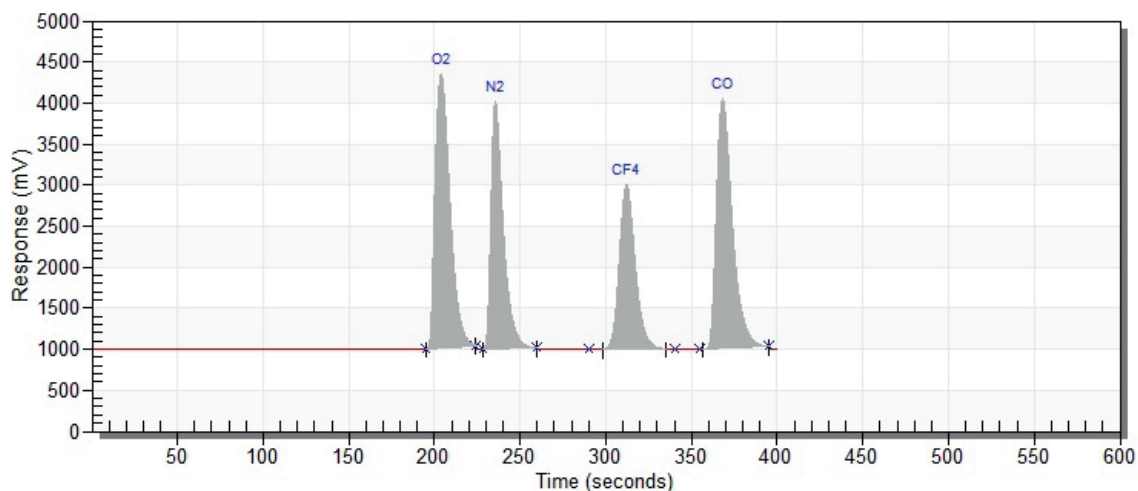


Figure 1: Chromatogram of trace impurities in balance SF6

COMPONENT	CONCENTRATION	PEAK HEIGHT	NOISE	LDL (3X NOISE)
O ₂	1701 ppm	4425 mV	2 mV	2.3 ppm
N ₂	1446 ppm	4101 mV	0.2 mV	0.6 ppm
CF ₄	64 ppm	3055 mV	10 mV	0.6 ppm
CO	196 ppm	4170 mV	5 mV	0.7 ppm

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

Figure 2: LDL based on 3 times noise ratio

CONCLUSION:

Using a solution combining the PlasmaDetek 2 and the MultiDetek 2 is the best way to achieve sensitivity, robustness and speed in a compact GC system for sulfur hexafluoride purity analysis for the energy market.



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APPLICATION NOTE

LD16-05



Refinery gas analyses with MultiDetek2 compact gas chromatograph and PlasmaDetek2 gas detector



The analysis of trace permanent gases has many different fields of application in the petrochemical industry. One of the most important is for controlling the manufacturing process and the product quality. For example, some contaminants as carbon monoxide and carbon dioxide tend to deteriorate the catalysts in the propylene and ethylene polymer grade production.

An instrument for monitoring trace impurities is then required. Many different GC techniques are available on the market. Most of the techniques use a combination of TCD, FID and methanizer for the trace analysis of H₂-O₂-N₂-CH₄-CO-CO₂ in propylene and ethylene. More precisely, an FID and a methanizer are used to trace CH₄-CO and CO₂. A TCD with Hydrogen or Helium carrier gas is used to trace O₂-N₂ detection. Finally, a second TCD with Argon or Nitrogen carrier gas must be added to trace H₂ detection. These solutions require complex GC solutions with multiple detectors and multiple gas sources for carrier, fuel and air. On top of that, an FPD must be added in some cases when the trace analysis of H₂S is required.

LDETEK SOLUTION:

The MultiDetek2 compact GC solution combined with the PlasmaDetek2 (PED) can perform the analysis of low concentration H₂-O₂-N₂-CH₄-CO-CO₂ in different petrochemical gases as propylene, ethylene, propane, butylenes, butane and some others.

This solution offers the advantage of having a single detection technology based on plasma emission detector to achieve detection limits from ppb to ppm as required for this type of application. A single carrier gas source is necessary. It can be Helium or Argon depending on the availability of gases on site.

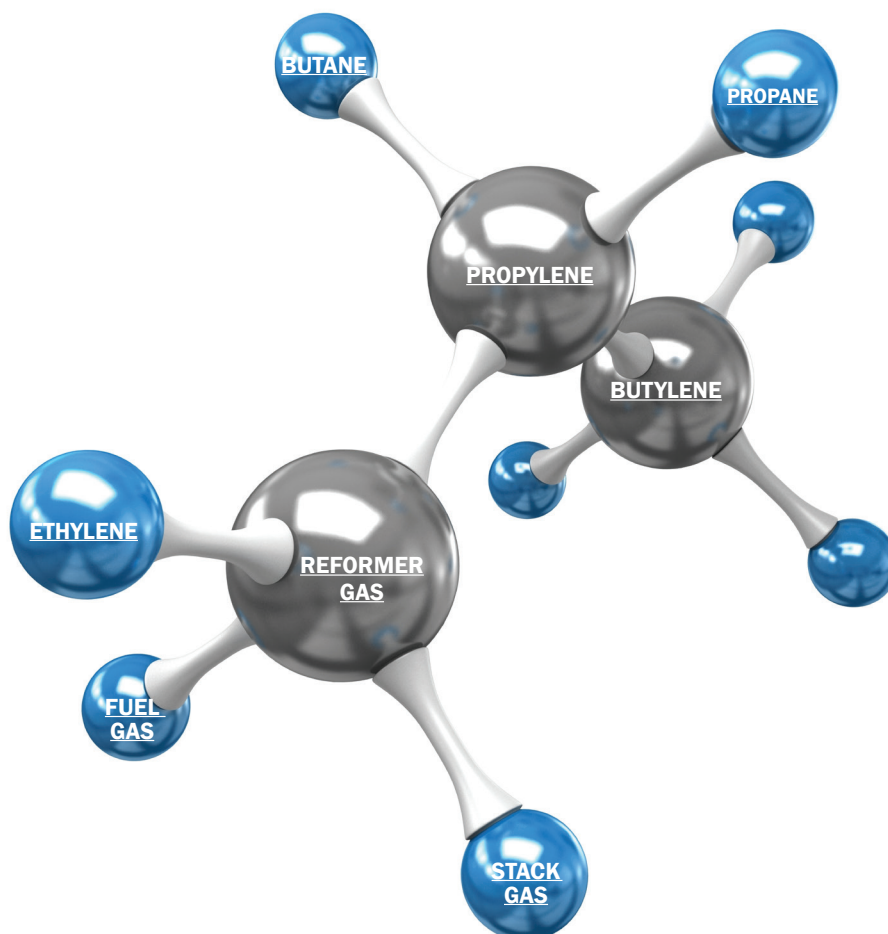
The method is configured with 2 channels merging in one PlasmaDetek2(PED). The first channel has a 10 port injection/backflush diaphragm valve with a Q Bond pre- column that allows the elution of H₂-O₂-N₂-CH₄-CO as one peak and then vent out the heavier gases. The second column is a molecular sieve used for the separation of the previously mentioned impurities that will go one by one to the PED through a diaphragm selection valve. The second channel will use the same hardware configuration as the first channel except that no molecular sieve column will be added. Then, after the elution of CO₂ through the pre- column, the heavier impurities as propylene, ethylene and others will be vented out of the system. Again, the CO₂ will be redirected to the PlasmaDete2 through the selection valve.

For some other RGA applications, it is also required to measure some light hydrocarbons and sulfurs. For these cases, the MultiDetek2 is so flexible that the configuration can be modified to achieve such measurement capability in the same compact chassis.

For example, the trace H₂S analysis can be added in the same channel as the one already used to trace CO₂ with the Q Bond column. No need to add extra detector as FPD or PFPD as generally used since the same PlasmaDetek2 can detect low concentration sulfurs. In the case that H₂S is added, then coated gas lines must be used to reduce surface adsorption. The detector doesn't have to be modified since it is made of Quartz, which is perfectly inert to trace sulfur.

For the light hydrocarbon analysis using the same MultiDetek2, a diaphragm valve network channel combined with Alumina type column and a second PlasmaDetek2 configured for hydrocarbons can be added. It is also possible to add an FID detector in the same unit if required.

The MultiDetek2 can be used in many different application fields related to the refinery gas analyses.



RESULTS:

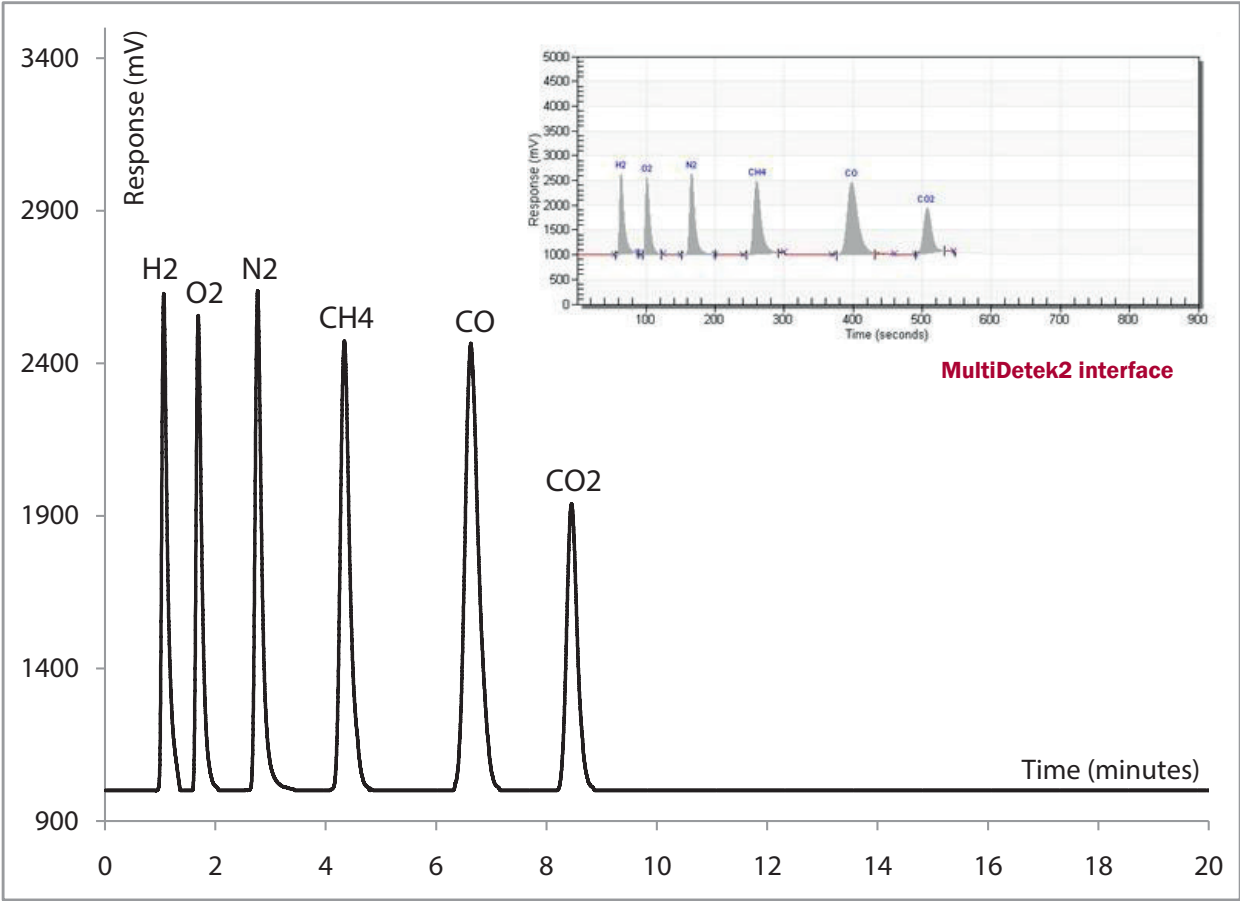


Figure 1: Chromatogram of trace impurities in balance Propylene

Historic Values

	H2	O2	N2	CH4	CO	CO2
0:31	8.103	9.177	10.531	9.687	9.675	5.186
7:05	8.090	9.163	10.532	9.680	9.578	5.193
3:39	8.140	9.185	10.545	9.674	9.544	5.198
0:14	8.113	9.184	10.537	9.657	9.578	5.171
4:03	8.044	8.981	10.532	9.657	9.475	5.188
0:37	8.155	9.197	10.564	9.695	9.578	5.198
7:11	8.155	9.209	10.586	9.694	9.695	5.113
3:46	8.253	9.129	10.596	9.668	9.578	5.138
0:19	8.232	9.163	10.655	9.638	9.535	5.198
5:53	8.233	9.226	10.612	9.602	9.544	5.226
3:28	8.017	9.163	10.564	9.628	9.475	5.193
0:02	8.265	9.271	10.545	9.602	9.475	5.233
5:35	8.218	9.267	10.579	9.644	9.578	5.198
3:10	8.155	9.129	10.564	9.657	9.696	5.236

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Figure 2: Results showing stability from a mixture cylinder containing trace impurities in balance gas Propylene

COMPONENT	CONCENTRATION	PEAK HEIGHT	NOISE	LDL (3X NOISE)
H ₂	8.155 ppm	2701 mV	2.2 mV	0.020 ppm
O ₂	9.129 ppm	2655 mV	1.5 mV	0.015 ppm
N ₂	10.564 ppm	2740 mV	1.1 mV	0.012 ppm
CH ₄	9.657 ppm	2501 mV	2 mV	0.023 ppm
CO	9.696 ppm	2482 mV	2.5 mV	0.029 ppm
CO ₂	5.236 ppm	2010 mV	2.1 mV	0.016 ppm

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

Figure 3: LDL based on 3 times noise ratio

CONCLUSION:

With this simple method, the MultiDetek2 compact and robust GC brings an innovative solution for this type of application. The platform also offers the standard industrial communication protocols, the remote connectivity and a built- in or external PC with software interface. The refinery gas analysis market can now have a compact, robust and flexible GC analyzer using a single detector type (PlasmaDetek2) and a single carrier type to cover the whole range of applications.



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APPLICATION NOTE

LD16-06



LD8000 MultiGas online gas analysis solution for high purity compressed Helium used in cryogenic installations.



▲ LD8000 MultiGas

▲ PlasmaDetek2

Cryogenics is the branch of physics that deals with the production and effects of very low temperatures. Helium was a natural choice of coolant as its properties allow components to be kept cool over long distances. Super fluid helium has remarkable properties, including very high thermal conductivity; it is an efficient heat conductor. These qualities make helium an excellent refrigerant for cooling and stabilising the LHC's large-scale superconducting systems. The Large Hadron Collider (LHC) at institutions like the CERN in Switzerland is the largest cryogenic system in the world and one of the coldest places on Earth. It is one of the examples where the use of cryogenic Helium is essential for good working of the system.

The difference from conventional industries is that repairing a cryogenic system and reaching the process nominal conditions is a time-consuming action due to the thermodynamic constraints. For this reason, fault diagnostic functions, capable to detect and identify faults before their degeneration into failures, become more and more important. In cryogenic installations, the most critical class of equipment consists of rotating machinery, such as pump, turbines and compressors. Beside the problems related to failures of compressors, turbines, etc... The limitation of plant runtime and failures are also due to impurity loads to the cold box. Mostly smaller sources of water and air contamination are found just before the beginning of appearance of the Breox oil in the vacuum screw compressors that leads to a high load of the cold box with water, formaldehyde and other organic components. This fault situation must absolutely be detected at the beginning to minimize the impact of failure.

This is why it is required to have an impurity monitoring instrument for the cold box feed gas that measures the ratios of air, water, hydrocarbons and oil in the gas running from the recycle compressor to the cold box. This way, the maintenance and actions can be taken at the right moment. The analytical instrument must monitor trace N₂-O₂-water and hydrocarbons in high purity Helium.

LDETEK SOLUTION:

LDetek offers its online LD8000 MultiGas based on plasma emission technology (PlasmaDetek2) to trace the O₂-N₂-CnHM and water impurities in cryogenic Helium. Using a single detection technique based on plasma emission, the trace impurities can be measured with an online mode. The required range for this type of application is usually 0-100ppm for O₂ and N₂, 0-25ppm for moisture and 0-10ppm for CnHM. Other ranges can be configured on request. The system monitors in continue the Helium purity without the use of Chromatography columns. The response analysis time for each gas can be done within 30 seconds. Better response time can be achieved depending on the purge flow rate that is adjustable on the LD8000. Since the Helium cost is an important factor, LDetek has designed its unit to ensure low sample flow consumption and this with consideration to ensure keeping a quick response time.

On top of that, the design is based on independent micro plasmas to ensure the protection of the system from oil contamination on long-term operation. A first plasma is isolated for measuring O₂ and N₂ together, having their own selective mode. A second plasma is used for measuring independently the H₂O concentration alone having its own flow path specifically designed for moisture analysis. And a third plasma combining a plasma converter system is used for measuring the trace CnHM. This third plasma system is designed to ensure that the optic used for measuring the CnHM isn't submitted to carbon deposit contamination.

FEATURES:

The instrument comes with a touchscreen interface and a keypad that facilitate the navigation through the different menus. It has one 4-20mA analog output per impurity. Each impurity has 2 ranges of operation and an individual ID range contact. It also has 2 configurable alarm contacts and one status contact. The unit has an automatic proportional valve for controlling the sample flow rate and a manual valve for adjusting the sample bypass flow rate.

Based on dual plasma operation, a safe mode has been implemented if the oil level goes over a certain concentration. It protects the alternative plasmas to be contaminated with carbons to guarantee a long-term operation in presence of dirty Helium gas containing high level of oil.

CONCLUSION:

The LD8000 MultiGas uses a parallel plasma system configuration that is individually selective to each measured gas to avoid the interferences from other impurities. Using this technique, the LD8000 becomes the right online instrument to be used for Helium purity for monitoring multiple impurities in a single unit on cryogenic installation.



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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.

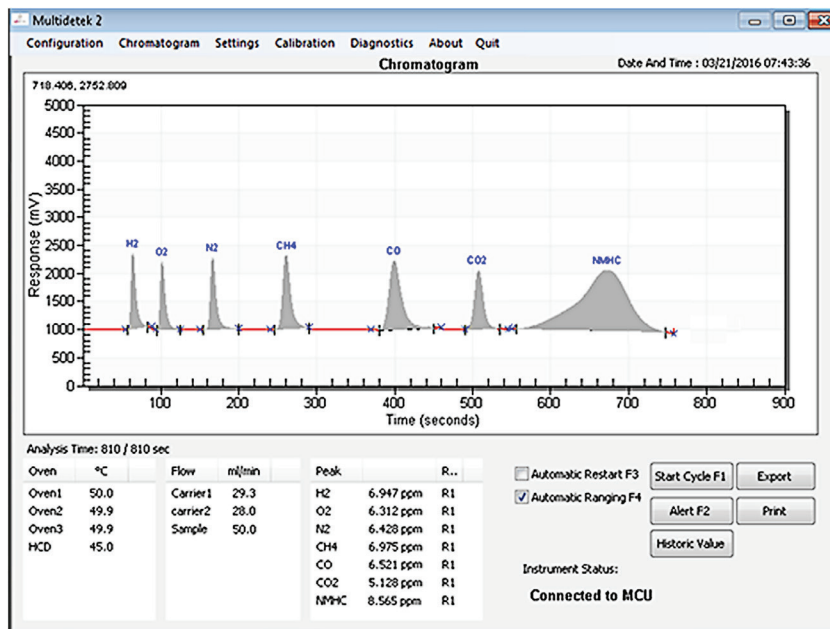


Figure 1

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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APPLICATION NOTE

LD16-08



Measurement of hydrocarbons in UHP Oxygen using the MultiDetek 2 and PlasmaDetek-E



▲ MultiDetek2

▲ PlasmaDetek2

This application note shows different methods that have been developed by LDetek for measuring the hydrocarbons in a stream of Oxygen (other matrixes can be analysed as well since the PlasmaDetek-E is strictly selective to hydrocarbons). This application note is the continuity of the application note LD16-02. It is then suggested to first read the LD16-02 to be advised about the technology that was used.

LDETEK SOLUTION:

The chromatograms shown in figure 1 and in figure 3 have been performed using a single column with a Plasmadetek-E. The system uses nitrogen as a unique carrier gas. The simplicity of this configuration makes this solution very efficient and simple for operation. No maintenance is required for such micro plasma based system.

Method for chromatogram in figure 1:

The MultiDetek2 configuration uses a 10 port diaphragm valve mounted in an injection/backflush mode. The sample gas is then introduced in the packed porous polymer type column for eluting the Air/O₂ followed by CH₄ in one direction to the detector before to reverse the flow in the column to get the NMHC redirected to the detector. The selective mode of the PlasmaDetek-E makes the detector being strictly selective to hydrocarbons and makes oxygen/air being invisible to the detector. The detector response and LDLs are demonstrated in figure 2. Please note that different performances can be achieved depending on the maximum and minimum ranges required.

Method for chromatogram in figure 3:

Using the same valve configuration combined with a different column type and system conditions, the analysis of separated C₂'s becomes possible. For some applications, where it is critical to measure C₂s individually, especially acetylene at low concentrations, this method is preferred. The addition of C₃+ measurement can be done in the same system to cover the whole hydrocarbon analysis solution. It offers better accuracy, sensitivity and simplicity than conventional FID or Argon plasma method. Figures 4-5 and 6 show an example of performances using this method in real life conditions.

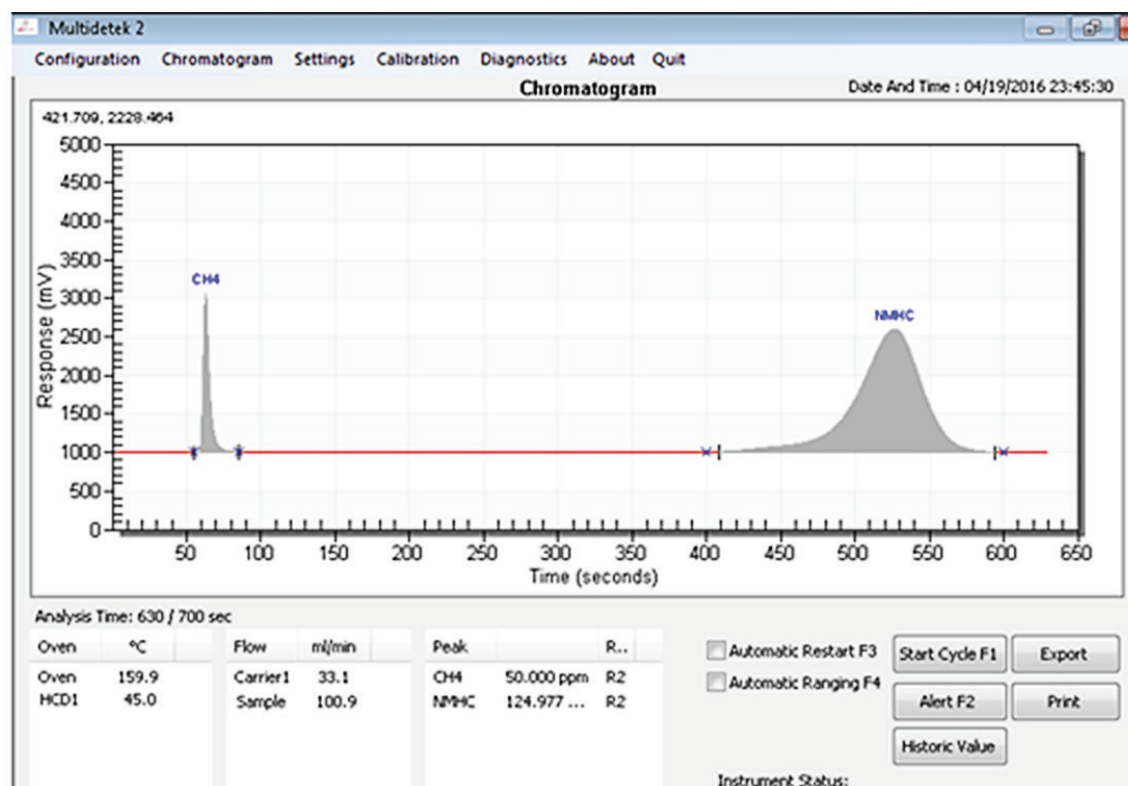


Figure 1: Chromatogram of CH₄ & NMHC in oxygen matrix

COMPONENT	CONCENTRATION	PEAK HEIGHT	NOISE	LDL (3X NOISE)
CH ₄	50.000 ppm	2211 mV	0.8 mV	0.054 ppm
NMHC	124.977 ppm	1622 mV	0.4 mV	0.092 ppm

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

Figure 2

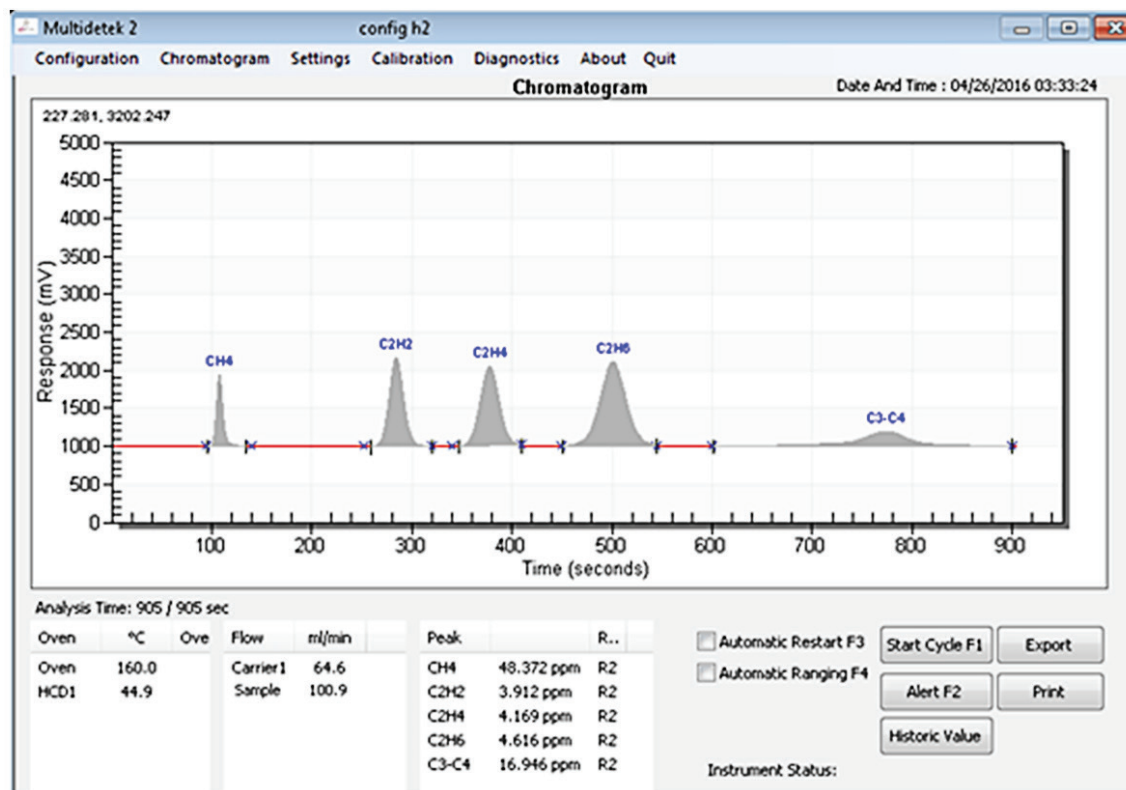


Figure 3: Chromatogram of trace CH₄, C₂H₂, C₂H₄, C₂H₆ & C₃+ in oxygen matrix

COMPONENT	CONCENTRATION	PEAK HEIGHT	NOISE	LDL (3X NOISE)
CH ₄	48.372 ppm	951 mV	0.3 mV	0.046 ppm
C ₂ H ₂	3.912 ppm	1241 mV	3.3 mV	0.031 ppm
C ₂ H ₄	4.169 ppm	1111 mV	3.3 mV	0.037 ppm
C ₂ H ₆	4.616 ppm	1146 mV	3.3 mV	0.040 ppm
C ₃ +	16.946 ppm	220 mV	0.9 mV	0.207 ppm

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

Figure 4

Time	CH4	C2H2	C2H4	C2H6	C3+
/2016 04:23:29	7.278	7.368	7.252	6.947	23.382
/2016 04:08:18	7.283	7.365	7.250	6.947	23.383
/2016 03:53:07	7.281	7.364	7.253	6.955	23.397
/2016 03:37:56	7.282	7.361	7.246	6.942	23.387
/2016 03:22:45	7.289	7.362	7.253	6.954	23.409
/2016 03:07:34	7.290	7.368	7.254	6.965	23.422
/2016 02:52:23	7.280	7.369	7.259	6.968	23.428
/2016 02:37:12	7.265	7.369	7.260	6.960	23.419
/2016 02:22:01	7.278	7.377	7.266	6.965	23.419
/2016 02:06:50	7.293	7.379	7.264	6.960	23.406
/2016 01:51:39	7.275	7.382	7.269	6.951	23.417
/2016 01:36:28	7.276	7.385	7.266	6.965	23.420
/2016 01:21:17	7.290	7.390	7.268	6.980	23.424
/2016 01:06:06	7.288	7.390	7.273	6.983	23.453

Figure 5: Example of stability results over 10 consecutive cycles

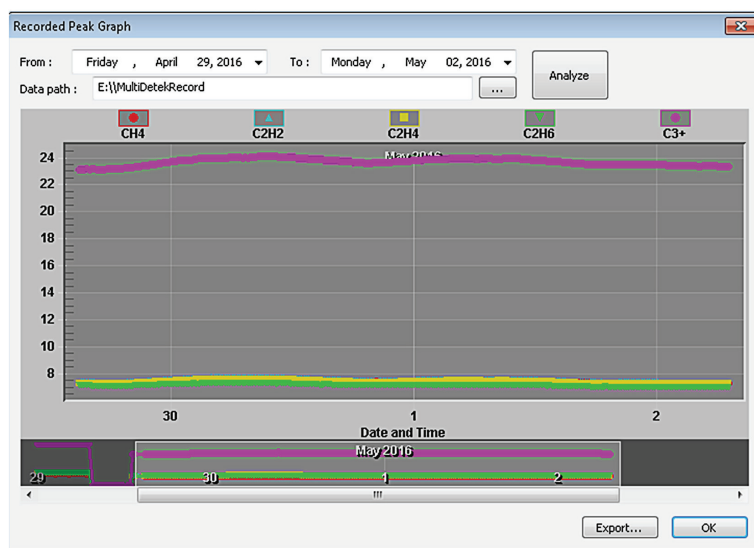


Figure 6: Example of stability results over a 3 day period

CONCLUSION:

The use of the PlasmaDetek-E in a compact MultiDetek2 gas chromatograph using nitrogen as carrier makes the hydrocarbon analysis being very simple compared to the other existing methods. Other variants of hydrocarbon combination, plus other impurities like CO₂-N₂O... can be measured with the use of this micro plasma based selective detection technique and the versatile MultiDetek2 design.



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APPLICATION NOTE

LD16-09



Measurement of trace impurities in UHP hydrogen



▲ MultiDetek2

▲ PlasmaDetek2

The production of UHP hydrogen requires analysis of trace impurities to control and certify the gas purity. Multiple instruments are usually dedicated to this task. Combining everything in the same instrument, the MultiDetek2 compact gas chromatograph is very efficient for this type of requirement. The detection down to part per billion can be achieved, what makes the instrument capable of certifying different hydrogen grades.

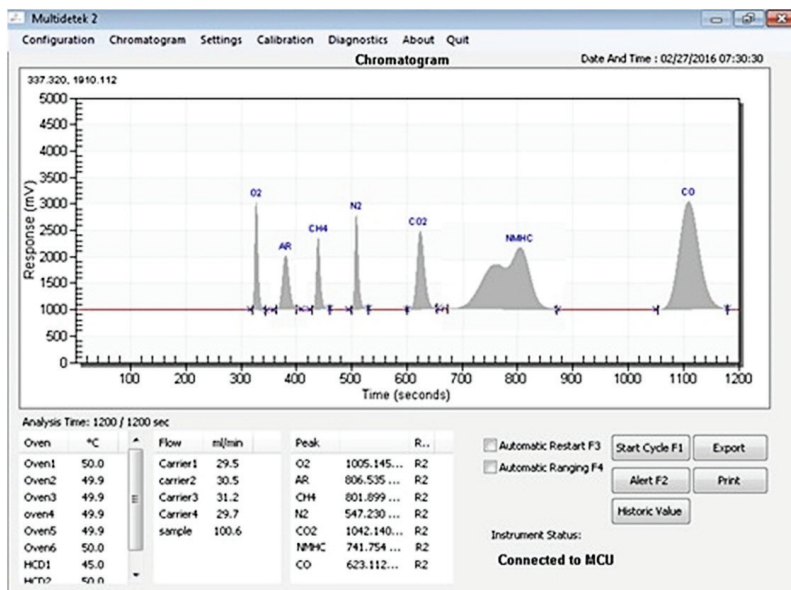
LDETEK SOLUTION:

Most of the gas chromatographs will use some combinations of hydrogen traps or membranes for measuring some impurities like Ar-O₂ and N₂ at low part per billion to eliminate the interference from hydrogen. The selectivity of the PlasmaDetek2 detector has the ability to measure those difficult impurities down to ppb level without the use of any hydrogen traps or membranes. It reduces the operation cost and simplifies the running operations of the system.

The configuration has 5 channels all converging in 3 different plasma detectors as follows:

- ▶ **Channel#1:** Measuring CH₄-CO₂ with HayeSep columns merging in plasma#1
- ▶ **Channel#2:** Measuring O₂-N₂ with Molecular Sieve columns merging in plasma #1
- ▶ **Channel#3:** Measuring NMHC with HayeSep column merging in plasma #2
- ▶ **Channel#4:** Measuring CO with Molecular Sieve column merging in plasma #2
- ▶ **Channel#5:** Measuring Ar with ArgoTek* column merging in plasma #3

*ArgoTek column is manufactured by LDetek to offer the trace argon separation from matrix Oxygen or Hydrogen. The column is packed type operating at 45 Celsius / 20 sccm helium carrier flow rate. With these conditions, the column offers the desired separation for measuring part per billion argon in either oxygen or hydrogen.



The three plasma detectors are respectively configured with the appropriate optical filters for blocking the hydrogen matrix and focussing on the dedicated impurities. This plasma configuration allows the analysis of multiple impurities in one single compact gas chromatograph unit.

Figures 1 and 2 show an example of results that such configuration can achieve for measuring low ppb/ppm concentrations of UHP hydrogen.

Figure 1: chromatogram of trace impurities in UHP hydrogen

COMPONENT	CONCENTRATION	PEAK HEIGHT	NOISE	LDL (3X NOISE)
O ₂	1005.1 ppb	2090 mV	2.1 mV	3.02 ppb
Ar	806.5 ppb	1017 mV	0.8 mV	1.90 ppb
CH ₄	801.8 ppb	1415 mV	1.6 mV	2.71 ppb
N ₂	547.2 ppb	1821 mV	1.4 mV	1.26 ppb
CO ₂	1042.1 ppb	1500 mV	2.2 mV	4.58 ppb
NMHC	741.7 ppb	1299 mV	2.2 mV	3.76 ppb
CO	623.1 ppb	2055 mV	5.1 mV	4.63 ppb

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

Figure 2

CONCLUSION:

The analysis of UHP hydrogen can be performed using this LDetek solution that combines a selective plasma detector configuration, a complete industrial compact gas chromatograph system, a unique separation column type with an impressive expertise in this field of application. Having all this makes LDetek solution perfect for this type of industrial application.



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APPLICATION NOTE

LD16-10



Measurement of trace Ar-Kr and N₂ in a bulk gas Oxygen



▲ MultiDetek2

▲ PlasmaDetek2

Detecting and measuring trace Ar-Kr and N₂ in a bulk gas Oxygen without the need of a cryogenic system, or a trapping adsorbent or an extra long column as generally used to measure trace ppb/ppm Argon as impurity from bulk Oxygen and for separating Krypton and Nitrogen.

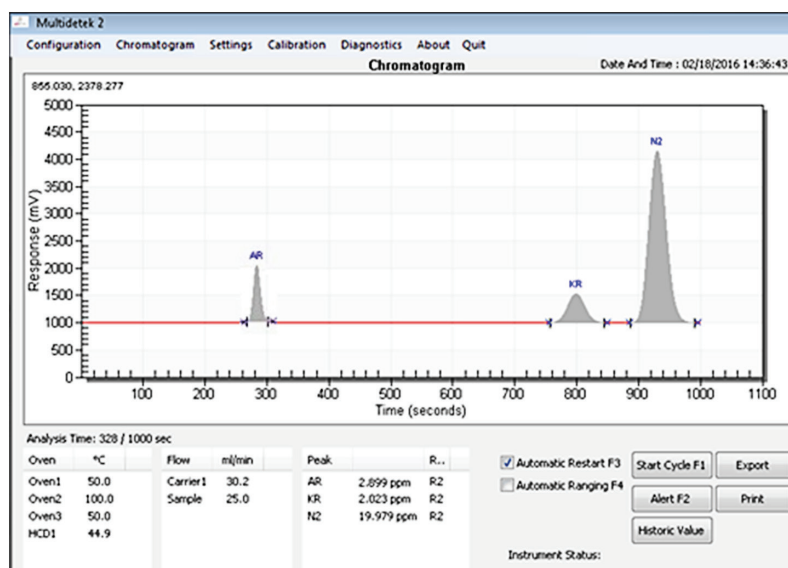
LDETEK SOLUTION:

Using the compact MultiDetek2 gas chromatograph, with a dual channel mode merging in one PlasmaDetek2 (PED) detector, the analysis of trace Ar-Kr and N₂ can be realized easily in a robust and maintenance free system.

- **Channel#1:** Used for measuring trace Argon in bulk Oxygen with the Argotek* column. The sample gas is injected using a standard 6 ports diaphragm valve directly in the packed type column that operates at isothermal temperature and with a fix carrier flow rate.
- **Channel#2:** Used for measuring trace Krypton and Nitrogen in bulk Oxygen with the HSR-Etek* column. The sample gas is injected using a standard 6 ports diaphragm valve directly in the packed type column that operates at isothermal temperature and with a fix carrier flow rate.

*ArgoTek column is manufactured by LDetek to offer the trace argon separation from matrix Oxygen. The column is packed type operating at 45Celsius/20sccm helium carrier flow rate. With these conditions, the column offers the desired separation for measuring part per billion argon in oxygen. (more details appear in application note LD12-3)

*HSR-Etek column is manufactured by LDetek to offer the trace Krypton and Nitrogen separation from matrix Oxygen. The column is packed type operating at 45Celsius/20sccm helium carrier flow rate. With these conditions, the column offers the desired separation for Kr/N₂. (more details appear in application note LD12-7)



Figures 1 and 2 show an example of results that such configuration can achieve for measuring low ppb/ppm concentrations in bulk oxygen.

Figure 1: chromatogram of trace impurities in Oxygen

COMPONENT	CONCENTRATION	PEAK HEIGHT	NOISE	LDL (3X NOISE)
Ar	2.899 ppm	1091 mV	0.7 mV	5.6 ppb
Kr	2.023 ppm	503 mV	0.8 mV	9.6 ppb
N2	19.979 ppm	3211 mV	0.7 mV	13.0 ppb

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

Figure 2

CONCLUSION:

The analysis of specific trace impurities in bulk Oxygen can be performed using this LDetek solution that combines a selective plasma detector with the right chromatography solution. It gives a robust and maintenance free system. Other impurities can be added to this system simply by adding extra channels.



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APPLICATION NOTE

LD16-11



Measurement of trace impurities in multiple bulk gases



▲ MultiDetek2

▲ PlasmaDetek2

Having an analytical system that is able to measure multiple impurities in different bulk gases is sometimes required. It is usually a big challenge to combine all the hardware in the same instrument.

LDETEK SOLUTION:

The MultiDetek2 (compact gas chromatograph)

This compact gas chromatograph can handle up to 3 detectors with 6 parallel chromatographic channels through a network combining up to 10 diaphragm valves and 12 chromatographic columns. It opens the doors to many applications requiring multiple bulk gases analysis using the same compact analytical instrument.

One configuration (method) is pre programmed for each bulk gas analysis requirement. This way, the user can simply load the desired configuration and the analyser is ready to start the analysis. As well, the impurities can be independently configured with specific ranges and minimum detection limits.

No external PC is required since the robust industrial PC is mounted in the instrument and gives access to a data storage capacity. The instrument can be controlled remotely through the Ethernet connectivity. All industrial communication protocols are accessible. Automatic or manual report printing can be handled.

The LDGSS (stream selector)

Multiple bulk gases analysis usually requires a stream selector system being able to be controlled locally or remotely. A manual switch is available on the front to select manually the desired stream. Otherwise, the stream can be selected via the MultiDetek2 interface. The interface gives the possibility to edit the names of the streams, to program multiple sequences and to set specific purging time between each stream. The streams can also be selected and identified by a standard DCS system through a dry contacts network.

This ultra high purity system offers a quick response time due to its dead volume free design. An adjustable sample purge valve and flow meter is independent to each stream to adjust the gas line purging before the stream selector. A purge gas being the same of the carrier gas of the MultiDetek2 is used to keep the ambient air away of the analytical flow path to ensure ultra high purity analysis gas for all the streams. Specifically when trace impurity of a bulk gas is also the pure gas of its neighbor stream that was just analysed in the previous analysis. The design of a back purged stream selector system is very important and this is what is included in this solution.

For this application note, since the analysis of hydrogen and oxygen is required, 2 LDGSS systems have been used to avoid mixing of both gases together. The purge gases are independent to each stream selector to avoid any chance of mixing oxygen and hydrogen. The LDGSS used for Oxygen can be certified for O2 Clean.

The COMPACT-LDP1000 (gas purifier)

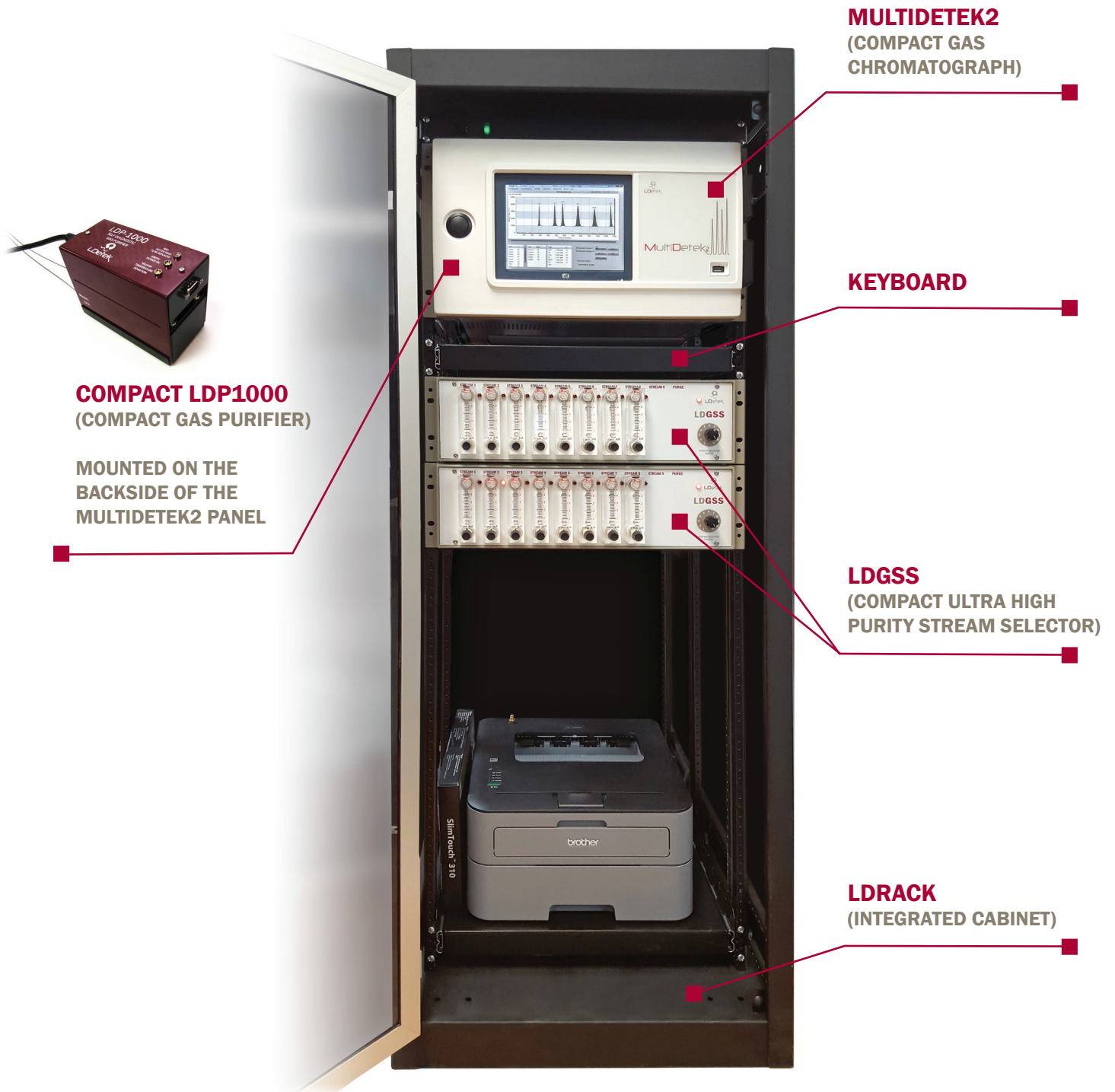
The gas purifier generates ultra high purity carrier gas certified for a total of 10ppb total impurities. This compact high capacity gas purifier keeps the system clean and intact even if carrier gas cylinders purity can vary from batch to batch introducing a variation of baselines which has impact on the accuracy of the analytical device. Or in another instance, if by mistake, an air contamination gets in the carrier flow path during the process of changing the carrier gas cylinder.

The gas purifier can also communicate in real-time with the MultiDetek2 to inform about his status. In the event of a default, it generates an alarm in the MultiDetek2 to automatically advise about the fault.

The LDRACK (certified cabinet)

The complete solution comes assembled and certified in a cabinet. Such system is fully tested to certify the analytical response time when switching between streams. This is more than necessary when low ppb analysis of nitrogen and oxygen are targeted. It doesn't only guarantee that the sample gas lines offers no dead volume and leaks, but also guarantees that the carrier gas lines are properly mounted. It is the best practice to get the best analytical performances especially when traces of impurity at ppb level are also present in ambient air must be detected.

THE COMPLETE SOLUTION



RESULTS:

Figures 1 to 12 show some examples of results that such configuration can achieve for measuring low ppb/ppm concentrations of multiple trace impurities in multiple bulk gases.

In this example, trace impurities H2-Ar-O2-N2-CH4-CO-CO2 in bulk gases Argon, Carbon dioxide, Hydrogen, Helium, Nitrogen and Oxygen were measured.

An optimized method for high range(HR) covering up to 1000ppm and another method for low range(LR) to ensure good peak detection and stability down to 10ppb are used. This dual methods technique gives the possibility to extend the dynamic range as desired. Conventional analysis techniques will tend to have some limitations on the low ppb detection when a high ppm analysis is required. The linearity and accuracy of our system is improved by having an optimized method for a specific analysis range.

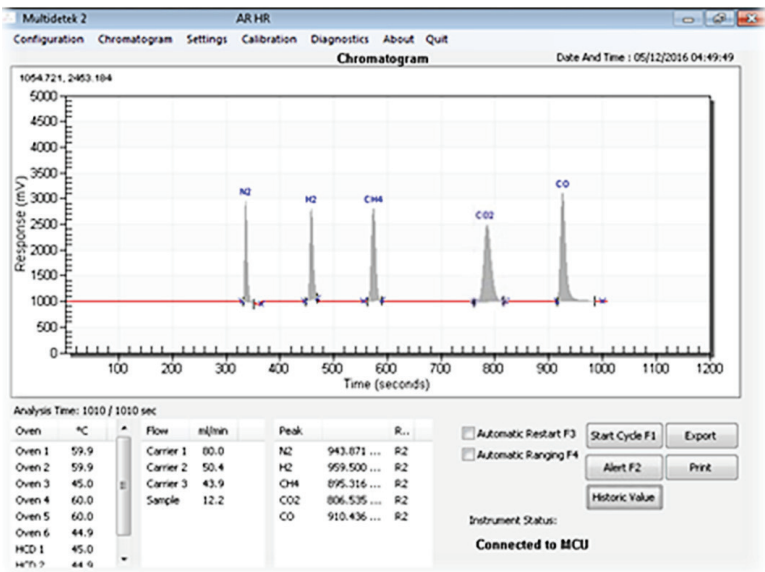


Figure 1:
chromatogram of trace impurities in Argon
(High range ppm)

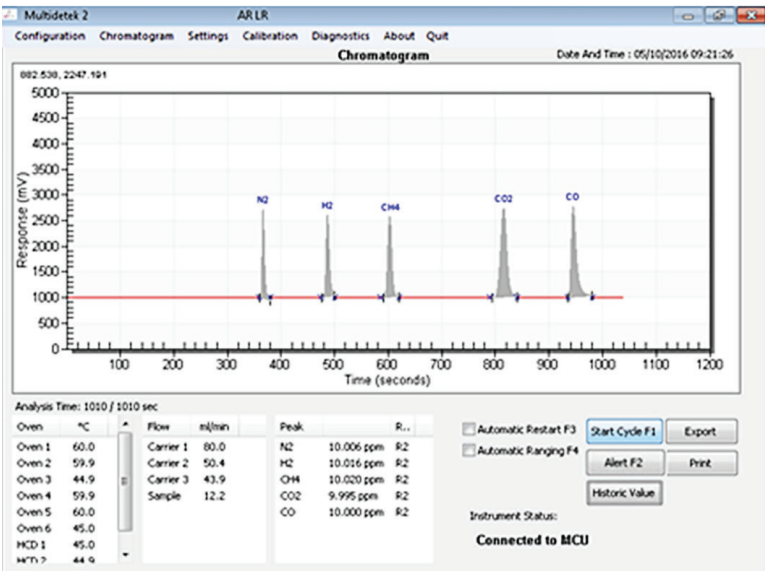


Figure 2:
chromatogram of trace impurities in Argon
(Low range ppb/ppm)

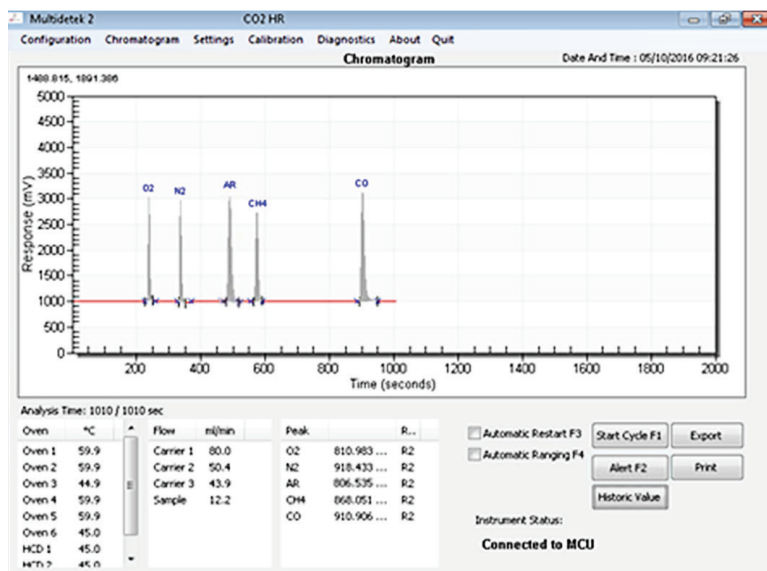


Figure 3:
chromatogram of trace impurities in Carbon Dioxide
(High range ppm)

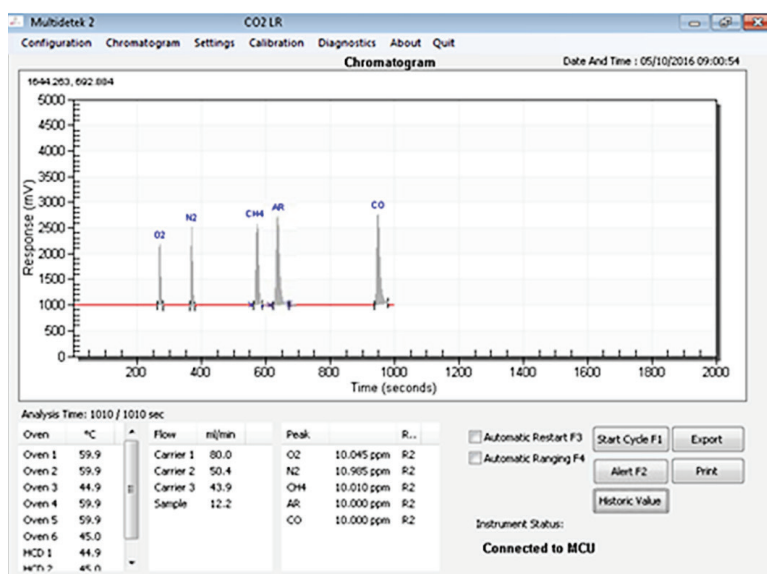


Figure 4:
chromatogram of trace impurities in Carbon Dioxide
(Low range ppb/ppm)

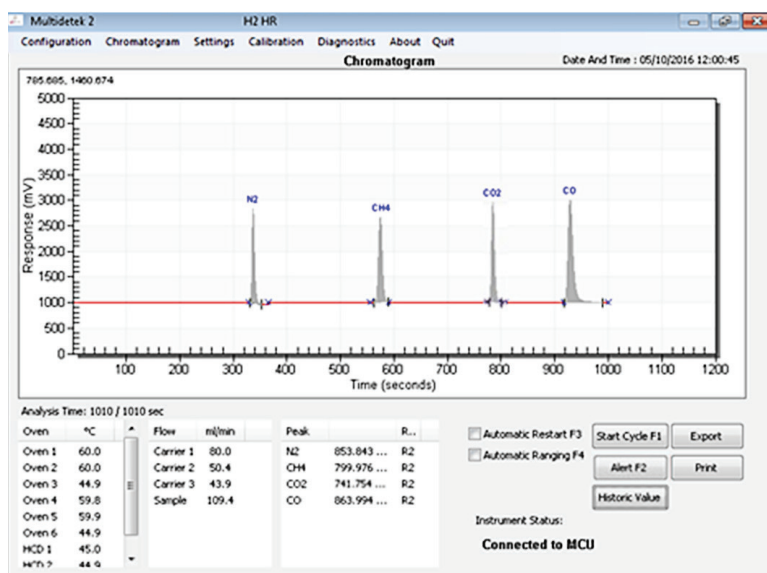


Figure 5:
chromatogram of trace impurities in Hydrogen
(High range ppm)

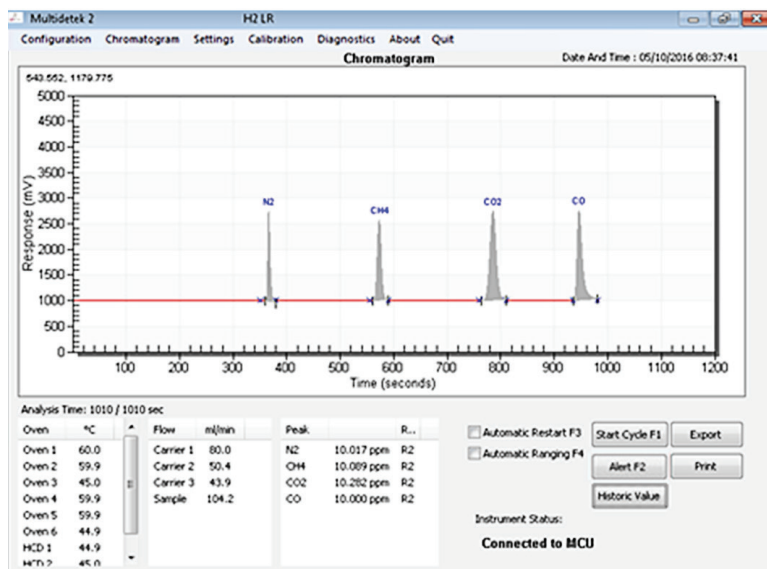


Figure 6:
chromatogram of trace impurities in Hydrogen
(Low range ppb/ppm)

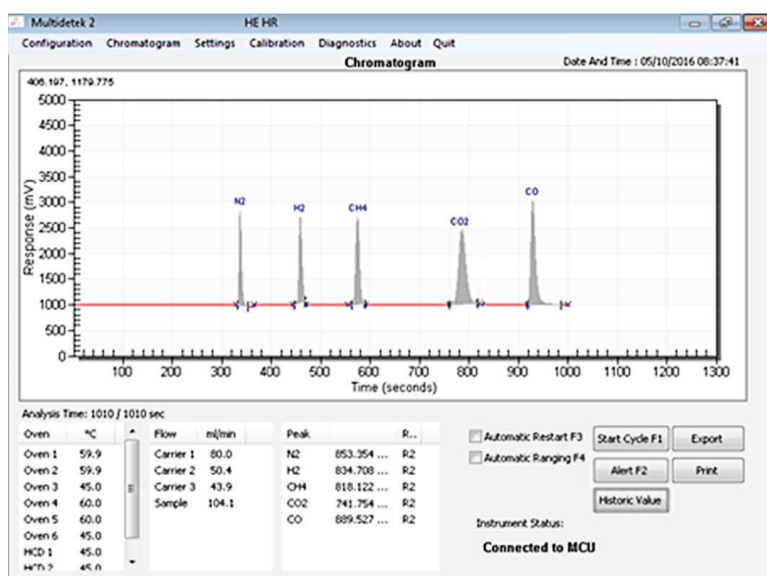


Figure 7:
chromatogram of trace impurities in Helium
(High range ppm)

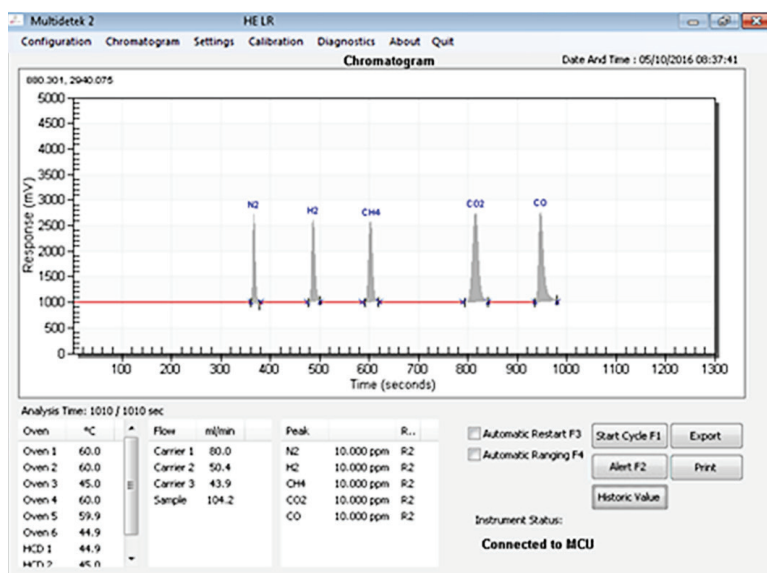


Figure 8:
chromatogram of trace impurities in Helium
(Low range ppb/ppm)

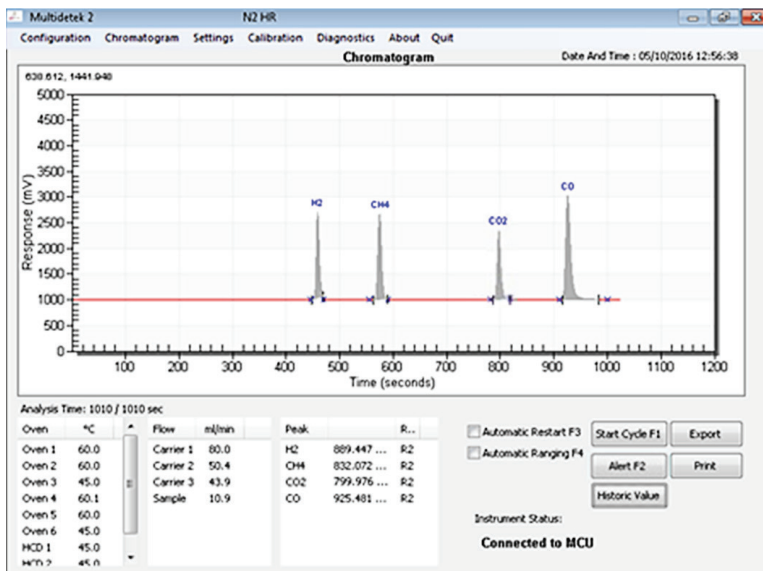


Figure 9:
chromatogram of trace impurities in Nitrogen
(High range ppm)

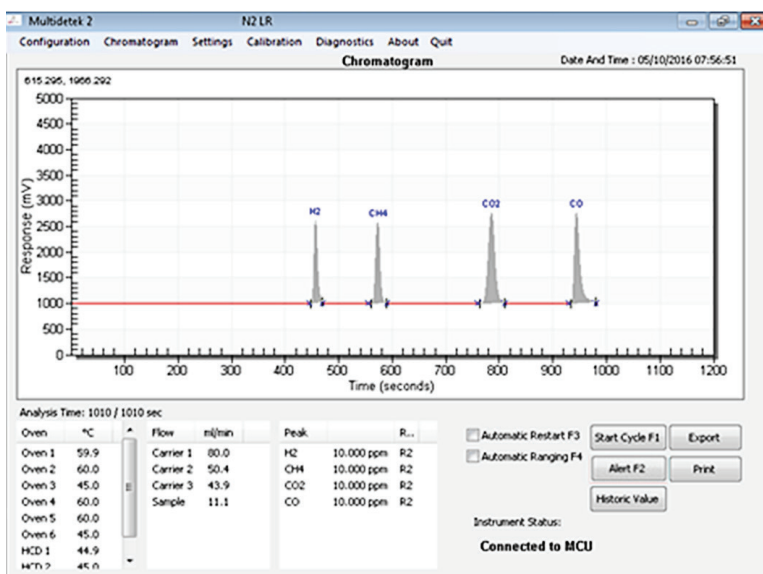


Figure 10:
chromatogram of trace impurities in Nitrogen
(Low range ppb/ppm)

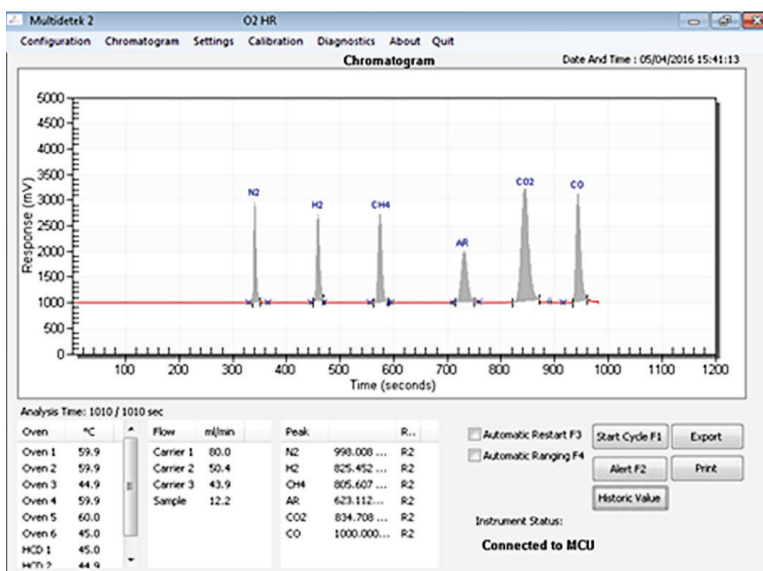


Figure 11:
chromatogram of trace impurities in Oxygen
(High range ppm)

COMPONENT	CONCENTRATION	PEAK HEIGHT	NOISE	LDL (3X NOISE)
H2 (fig.8)	10.000 ppm	1621 mV	0.7 mV	12.9 ppb
Ar (fig.4)	10.000 ppm	1780 mV	0.4 mV	6.7 ppb
O2 (fig.4)	10.045 ppm	1320 mV	0.6 mV	13.7 ppb
N2 (fig.2)	10.006 ppm	1821 mV	0.5 mV	8.2 ppb
CH4 (fig.6)	10.089 ppm	1612 mV	0.6 mV	11.3 ppb
CO (fig.10)	10.000 ppm	1806 mV	0.9 mV	14.9 ppb
CO2 (fig.6)	10.282 ppm	1823 mV	0.8 mV	13.5 ppb

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

Figure 12

CONCLUSION:

Our complete integrated gas analysis solution guarantees the best performances and robustness for any applications combining multiple impurities in multiple streams and/or bulk gas types. It is compact and compatible with standards of the industry.



Where **innovation** leads to **success**

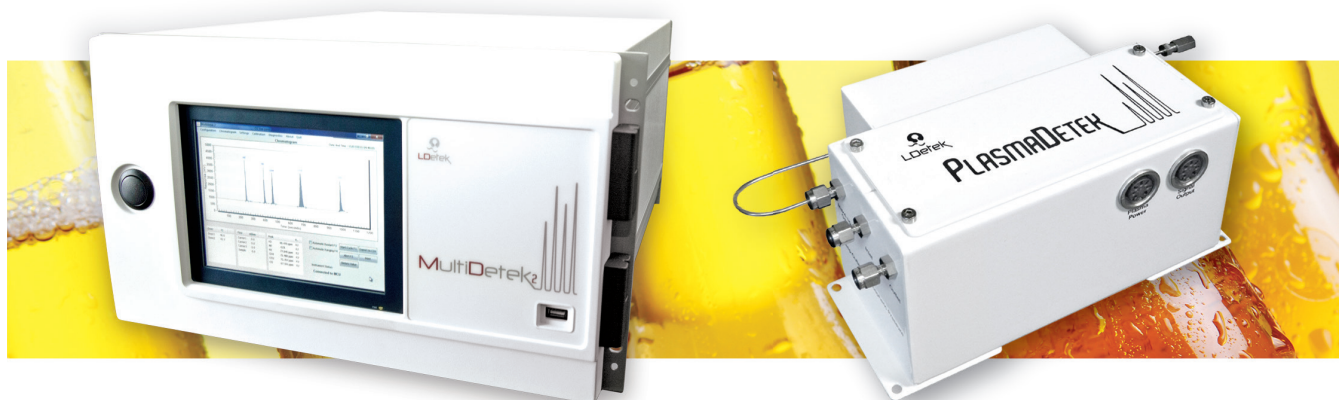
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APPLICATION NOTE

LD16-12



Trace impurities in Carbon Dioxide for beverage and food packaging industry



▲ MultiDetek2

▲ PlasmaDetek2

With regards to the beverage industry, the dissolved Carbon Dioxide which is used as carbonic acid gives a pleasantly acidic flavour and a nice mouth-feel when drinking. When it is not present, the drinks taste flat. Being used in many different fields of food and beverage, the CO₂ quality management is essential to meet the market requirements.

The CO₂ is produced from different techniques such as fermentation, combustion, ammonia/hydrogen production and others. It is required by the industry, especially for bottlers to control the supply chain by monitoring the CO₂ purity allowing maintenance of the product quality.

LDETEK SOLUTION:

Using the PlasmaDetek2(PED) plasma detector and the MultiDetek2 compact gas chromatograph, the analysis of the most critical trace impurities in carbon dioxide can be achieved in one unit with a single detection technology (PED).

By default, the configuration has 3 channels to cover the analysis of Benzene, Acetaldehyde, Nitrogen and Carbon Monoxide. If additional options for the analysis of Methanol, Sulfurs and Hydrocarbons are required, then channels 4, 5 and 6 are added in the same instrument.

Channel#1: 0-100ppb Benzene

Channel#2: 0-1000ppb Acetaldehyde

Channel#2: 0-120ppm Nitrogen & 0-10ppm Carbon Monoxide

Also integrated inside the MultiDetek2, a dilution system allows generating for a span calibration gas at ppb level for the low concentration impurities. This option gives you the capacity to use a standard certified calibration gas at higher concentrations, which is usually lower in cost and easier to get, also that generates a low ppb calibration gas. The dilution system is designed for low gas consumption, high accuracy/stability and is automatically handled by the interface during span calibration process.

For multiple streams requirement, the LDGSS high purity stream selector system can be combined with the MultiDetek2 to offer all the flexibility to switch streams and even run stream sequences analysis as required.

RESULTS:

The results show the performances of the system for a standard configuration used to measure Benzene, Acetaldehyde, Nitrogen and Carbon Monoxide in Carbon Dioxide.

It displays details about the calibration chromatogram, some examples of low ppb chromatograms for each impurity, the IdI chart for noise/response calculation, the stability and the linearity curves.

SPAN CALIBRATION CHROMATOGRAM:

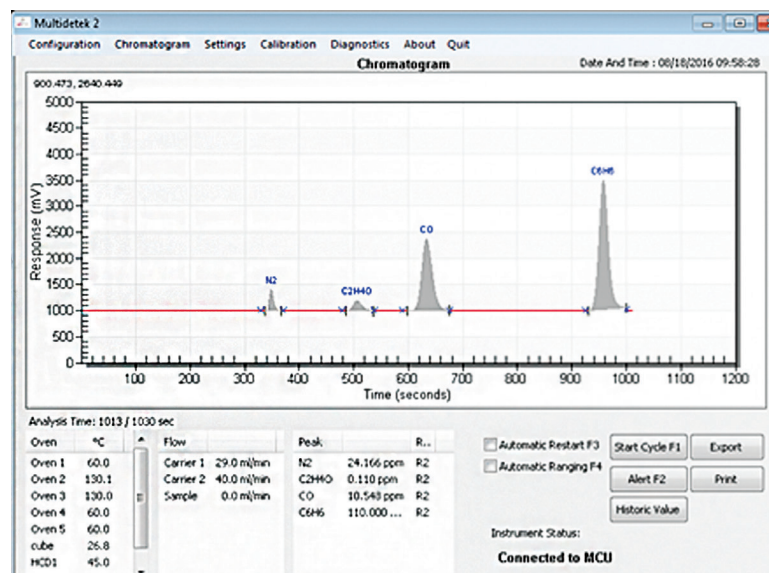


Figure 1:
Chromatogram of a gas mixture containing 24ppm nitrogen, 110ppb acetaldehyde, 10.5ppm carbon monoxide and 110ppb benzene in a balance gas carbon dioxide.

LOW PPB CHROMATOGRAM:

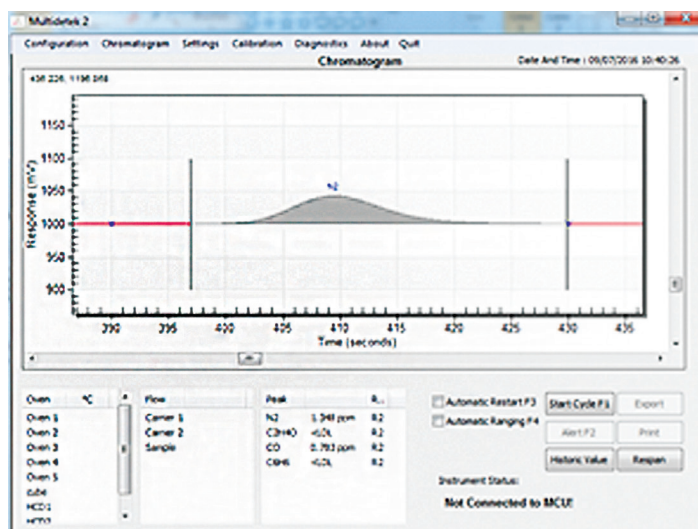


Figure 2:
Chromatogram of 1348 ppb Nitrogen in Carbon dioxide

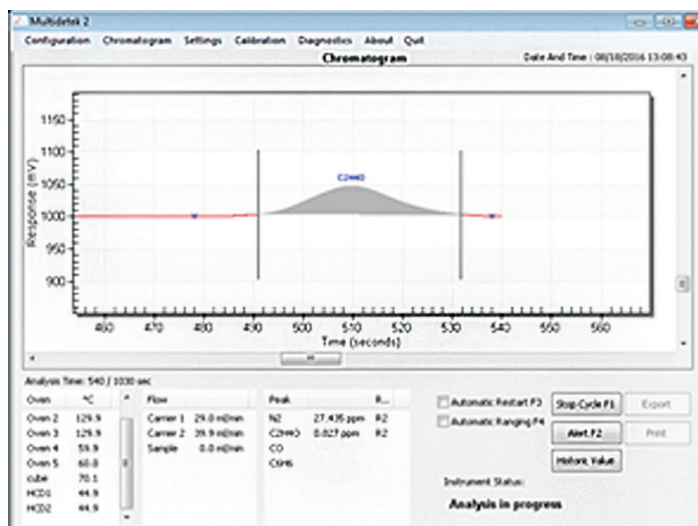


Figure 3:
Chromatogram of 27ppb Acetaldehyde in Carbon dioxide

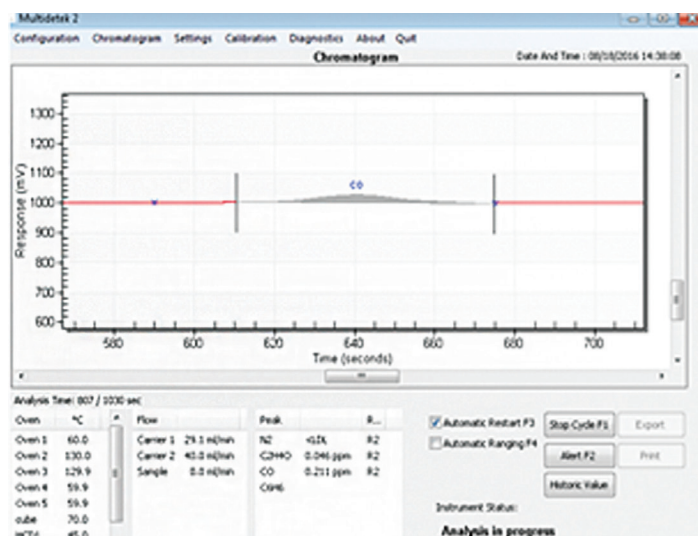


Figure 4:
Chromatogram of 211ppb Carbon monoxide in Carbon dioxide

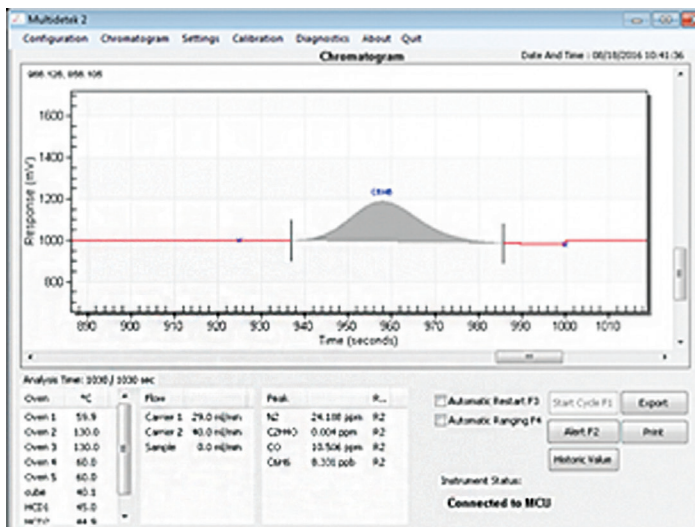


Figure 5:
Chromatogram of 8.331ppb Benzene in Carbon dioxide

LDL calculation

COMPONENT	CONCENTRATION	PEAK HEIGHT	NOISE	LDL (3X NOISE)
Nitrogen	1348 ppb	49.3 mV	0.2 mV	16.4 ppb
Acetaldehyde	27 ppb	49.1 mV	1.1 mV	1.8 ppb
Carbon monoxide	211 ppb	35.4 mV	1.6 mV	28.6 ppb
Benzene	8.331 ppb	215.7 mV	2.1 mV	0.243 ppb

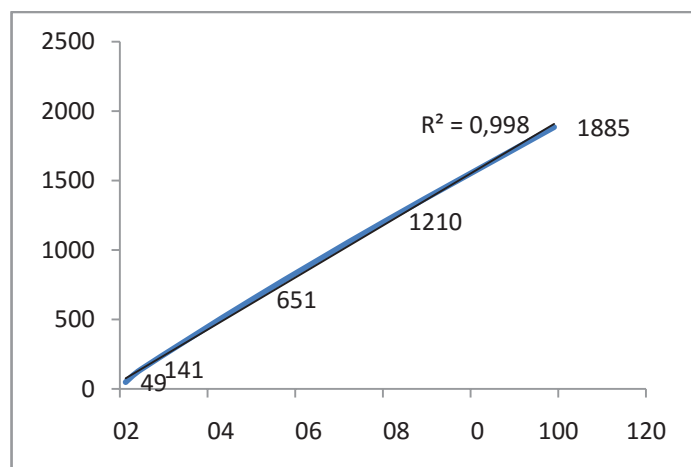
Note: other LDL could be obtained with different injection volume and chromatographic conditions

STABILITY:

Date/Time	N2	C2H4O	CO	C6H6
Aug/18/2016 08:07:47	24.596	0.783	10.601	57.229
Aug/18/2016 07:50:32	24.678	0.783	10.601	57.165
Aug/18/2016 07:33:17	24.606	0.784	10.597	57.110
Aug/18/2016 07:16:00	24.588	0.784	10.615	57.026
Aug/18/2016 06:58:45	24.579	0.784	10.613	57.102
Aug/18/2016 06:41:29	24.617	0.783	10.613	56.838
Aug/18/2016 06:24:13	24.768	0.782	10.617	56.933
Aug/18/2016 06:06:57	24.742	0.781	10.617	56.960
Aug/18/2016 05:49:41	24.627	0.780	10.601	56.890
Aug/18/2016 05:32:25	24.720	0.780	10.599	56.752
Aug/18/2016 05:15:10	24.687	0.780	10.599	56.860
Aug/18/2016 04:57:54	24.762	0.780	10.596	56.920
Aug/18/2016 04:40:38	24.797	0.780	10.599	56.879
Aug/18/2016 04:23:22	24.827	0.780	10.599	57.006
Aug/18/2016 04:06:06	24.728	0.780	10.604	56.974

Figure 6:
The results show the stability over a period of 15 consecutive cycles. The units for nitrogen, acetaldehyde and carbon monoxide are ppm and the unit for benzene is ppb

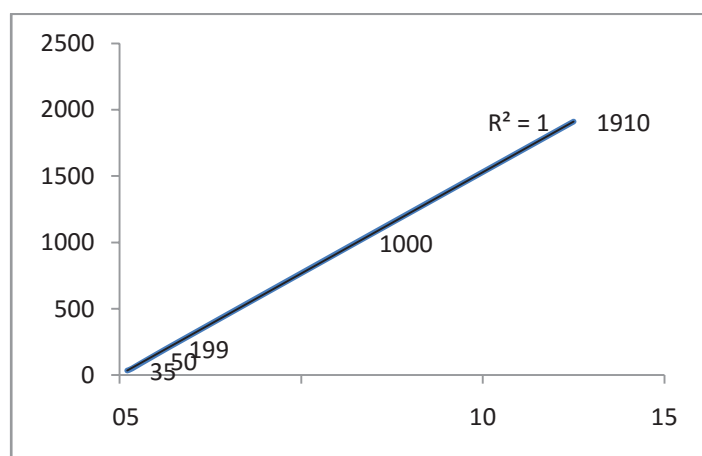
LINEARITY:



N2 CONCENTRATION N2 RESPONSE

1.348 ppm	49 mV
4.856 ppm	141 mV
30.518 ppm	651 mV
60.6 ppm	1210 mV
99 ppm	1885 mV

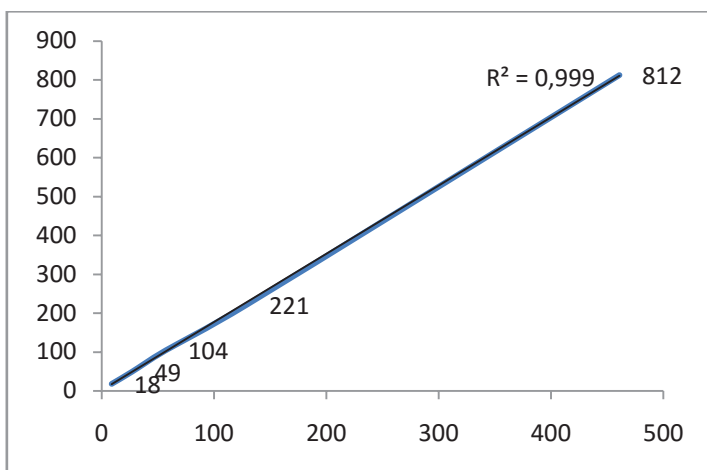
Figure 7:
Impurity : nitrogen



CO CONCENTRATION CO RESPONSE

0.211 ppm	35 mV
0.343 ppm	50 mV
1.285 ppm	199 mV
6.521 ppm	1000 mV
12.5 ppm	1910 mV

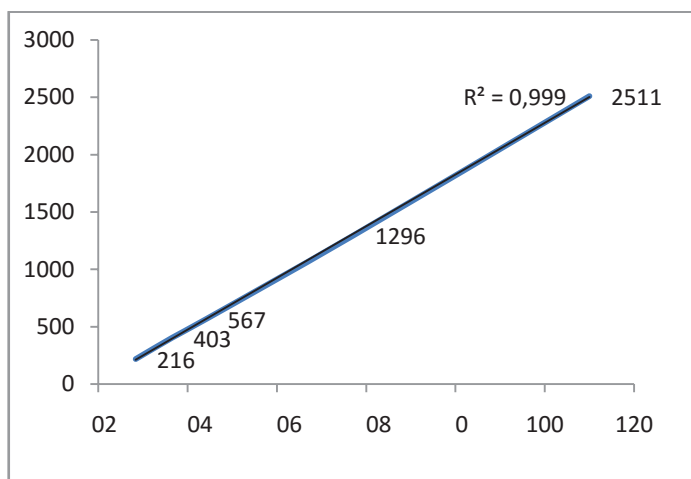
Figure 8:
Impurity : carbon monoxide



ACETALDEHYDE CONCENTRATION ACETALDEHYDE RESPONSE

9 ppb	18 mV
27 ppb	49 mV
57 ppb	104 mV
129 ppb	221 mV
461 ppb	812 mV

Figure 9:
Impurity : acetaldehyde



BENZENE CONCENTRATION	BENZENE RESPONSE
8.331 ppb	216 mV
16.532 ppb	403 mV
24.212 ppb	567 mV
57.158 ppb	1296 mV
110 ppb	2511 mV

Figure 10:
Impurity : benzene

CONCLUSION:

Our solution detects traces of gas impurities required by the food and beverage industry for Carbon Dioxide. The complete spectrum analysis can be covered with one single MultiDetek2 unit using the PlasmaDetek2 detection technology. It can detect sub ppb concentrations that are required for this type of application. It combines the analysis of different gas types that are usually very difficult to do with the same instrument. The MultiDetek2 offers a solution with an integrated PC interface that offers standard communication protocols compatible with all market standards.



Where **innovation** leads to **success**

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APPLICATION NOTE

LD17-01



Trace Hydrocarbons and Permanent gases in Propylene



The high purity Propylene is used for the production of Polypropylene in Petrochemical industry. The analysis of trace impurities is critical to ensure a good quality of the final product. The analysis of hydrocarbons and permanent gases are required at a level below 10ppm to ensure the good operation of the production process.

LDETEK SOLUTION:

Using the PlasmaDetek2(PED) plasma detector and the MultiDetek2 compact gas chromatograph, the analysis of the most critical trace impurities in Propylene can be achieved in one unit with a single detection technology (PED).

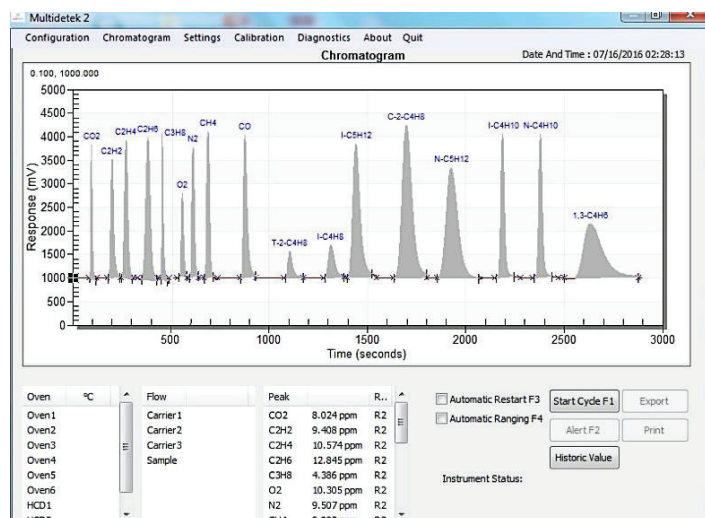
The main advantage of our solution is to use the same PlasmaDetek2 (PED) detector to measure trace impurities hydrocarbons and permanent gases in the same system. Usually, the alternative solutions have to combine more than one detector to be able to cover this application. Typically, FID and PDHID have to be combined, resulting of multi gas feeding for the different detectors. Our solution requires only one PED detector with only carrier gas to feed the system.

Our configuration has 4 channels having each an independent oven/column. The four channels all merge in the same PlasmaDetek2 detector. The detector is optimized with a combination of selective optic circuits especially for each impurity. This allows a good sensitivity and selectivity over the propylene background gas.

- ▶ **Channel#1:** Trace O₂, N₂, CH₄, CO
- ▶ **Channel#2:** Trace CO₂, C₂H₂, C₂H₄, C₂H₆, C₃H₈
- ▶ **Channel#3:** Trace i-C₄H₁₀, n-C₄H₁₀
- ▶ **Channel#4:** Trace t-2-C₄H₈, i-C₄H₈, i-C₅H₁₂, C-2-C₄H₈, n-C₅H₁₂, 1.3-C₄H₆

RESULTS

The chromatogram below shows an example of a typical calibration containing trace impurities in a balance gas of pure propylene. The concentrations of each impurities along with the response and detection limit are listed in the LDL chart below.



COMPONENT	CONCENTRATION	PEAK HEIGHT	NOISE	LDL (3X NOISE)
CO ₂	8.024 ppm	2820 mV	2.2 mV	18.8 ppb
C ₂ H ₂	9.408 ppm	2556 mV	2.2 mV	24.2 ppb
C ₂ H ₄	10.574 ppm	2899 mV	2.2 mV	24.0 ppb
C ₂ H ₆	12.845 ppm	3009 mV	2.2 mV	28.2 ppb
C ₃ H ₈	4.386 ppm	3086 mV	2.9 mV	12.3 ppb
O ₂	10.305 ppm	1770 mV	0.9 mV	15.7 ppb
N ₂	9.507 ppm	2758 mV	1.1 mV	11.4 ppb
CH ₄	9.892 ppm	3176 mV	2.2 mV	20.5 ppb
CO	9.122 ppm	3096 mV	2.8 mV	24.7 ppb
T-2-C ₄ H ₈	2.165 ppm	578 mV	2.2 mV	24.7 ppb
I-C ₄ H ₈	2.311 ppm	764 mV	2.2 mV	19.9 ppb
I-C ₅ H ₁₂	8.887 ppm	2865 mV	2.2 mV	20.5 ppb
C-2-C ₄ H ₈	9.102 ppm	3643 mV	2.2 mV	16.5 ppb
N-C ₅ H ₁₂	7.994 ppm	2424 mV	2.2 mV	21.8 ppb
I-C ₄ H ₁₀	9.111 ppm	3110 mV	2.2 mV	19.3 ppb
N-C ₄ H ₁₀	9.291 ppm	3121 mV	2.2 mV	19.6 ppb
1,3C ₄ H ₆	4.100 ppm	1256 mV	2.2 mV	21.5 ppb

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

CONCLUSION:

Our solution combining a PlasmaDetek2 (PED) with a compact GC MultiDetek2 is simple and robust for this type of application required by the market. Our solution also includes the standard industrial communication protocols to control the unit.



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APPLICATION NOTE

LD17-02



Gas analysis for wineries



In the wineries for proper wine production, inert gases like nitrogen, carbon dioxide and argon are used for sparging, blanketing as counter-pressure to move wine(usually from barrels), as well as to flush transfer lines and tanks prior to moving wine or juice. Sparging involves the introduction of a stream of very fine gas bubbles to help add or remove dissolved Oxygen or CO₂. Blanketing partially-filled tanks attempts to maintain an inert gas layer above the wine/ juice surface in the hope of minimizing wine/air contact. The main reason for the use of inert gassing is to prevent the growth of aerobic microorganisms in the wine. The gas chromatography is a well known analysis technique to ensure the measurement of the purity and to control the production of wine to achieve the best quality.

LDETEK SOLUTION:

Using the PlasmaDetek2(PED) plasma detector and the MultiDetek2 compact gas chromatograph, the analysis of the most critical impurities in carbon dioxide, nitrogen and argon used in wine production can be achieved with a single unit.

The configuration uses one channel for trace analysis of O₂-N₂-CO₂-Ethanol with the PlasmaDetek2(PED) optimized for low concentrations. The second channel is configured with a conventional TCD optimized for high concentrations in % for O₂-N₂-CO₂-Ethanol. This dual detectors configuration allows covering a wide analysis range.

- **Channel#1:** Trace O₂-N₂-CO₂-Ethanol
- **Channel#2:** % O₂-N₂-CO₂-Ethanol

Since the sample volume and pressure for this type of application is limited, the MultiDetek2 is equipped with a mini pump that sucks the right amount of sample to fill the sampling loops correctly prior to run the analysis. The parameters can be adjusted by the user easily from the software interface depending of the sample line selected. The sample collection system is designed(leak free) for low O₂-N₂ analysis performances without air contamination.

The advantages of our solution over standard chromatograph systems is based on the use of Argon as carrier gas with the PlasmaDetek2. It allows an easy GC configuration that can measure O₂-N₂ with a single column configuration, having low operating cost and getting good sensitivity at ppb level. This isn't feasible with standard ionisation detector because of the use of Helium as carrier gas, the O₂ analysis becomes not possible and the operating cost are higher.

RESULTS

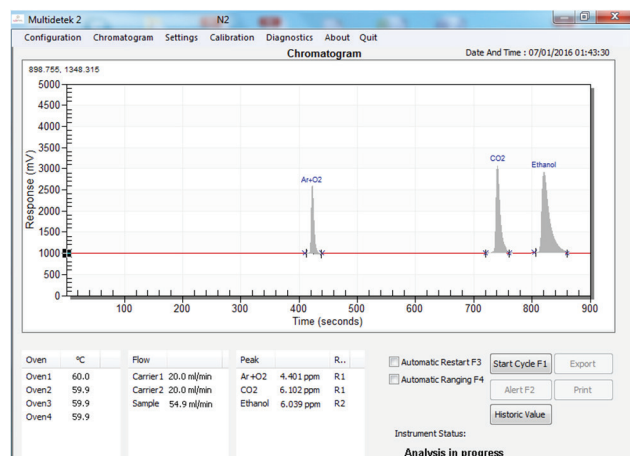
The results show the performances of the system for each pre configured method used to measure impurities in each sample type.

Method #1 is configured and calibrated for trace (Ar+O₂)-CO₂-ethanol in sample gas nitrogen.

Method #2 is configured and calibrated for trace N₂-CO₂-ethanol in sample gas argon.

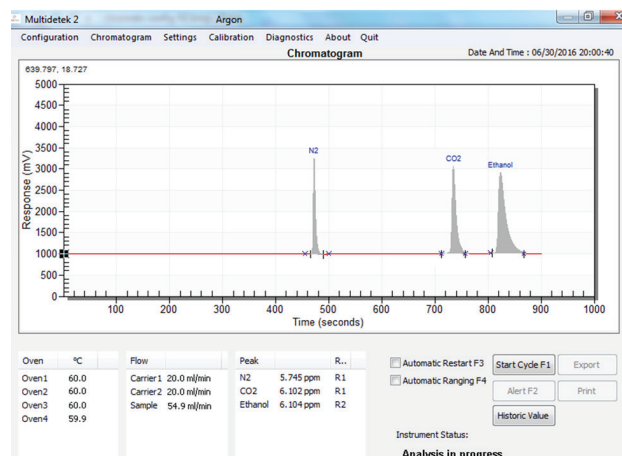
Method #3 is configured and calibrated for percent (Ar+O₂)-N₂-ethanol in sample gas carbon dioxide.

Span calibration chromatogram for Nitrogen method:



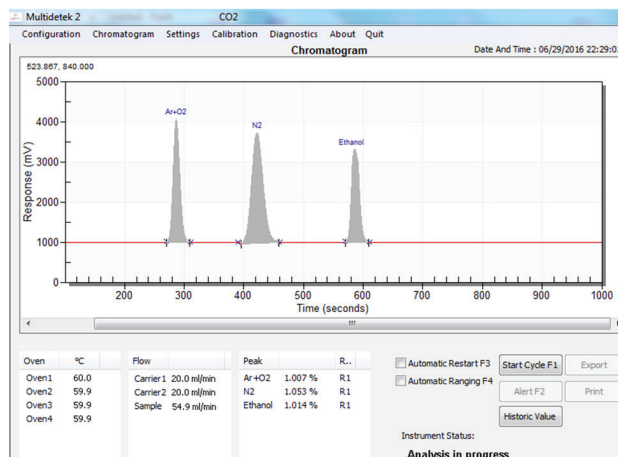
Chromatogram of a gas mixture containing 4.4ppm O₂, 6.1ppm CO₂ and 6.0ppm ethanol in a balance gas Nitrogen.

Span calibration chromatogram for Argon method:



Chromatogram of a gas mixture containing 5.7ppm N₂, 6.1ppm CO₂ and 6.1ppm ethanol in a balance gas Argon.

Span calibration chromatogram for Carbon dioxide method:



Chromatogram of a gas mixture containing 1% O₂, 1% N₂ and 1% ethanol in a balance gas Carbon dioxide.

CONCLUSION:

Our solution combines our PlasmaDetek2 for low ppb/ppm concentrations and a TCD for the percent concentrations. Having this dual detectors configuration, a wide range of analysis can be covered. The MultiDetek2 is configured with multiple methods depending of the requirements. It is easy for the user to simply load the proper method for his requirement. On request, additional impurities and methods can be added to the same system.

Our solution is robust and perfectly adapted for the gas analysis used in the wine production sector.



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