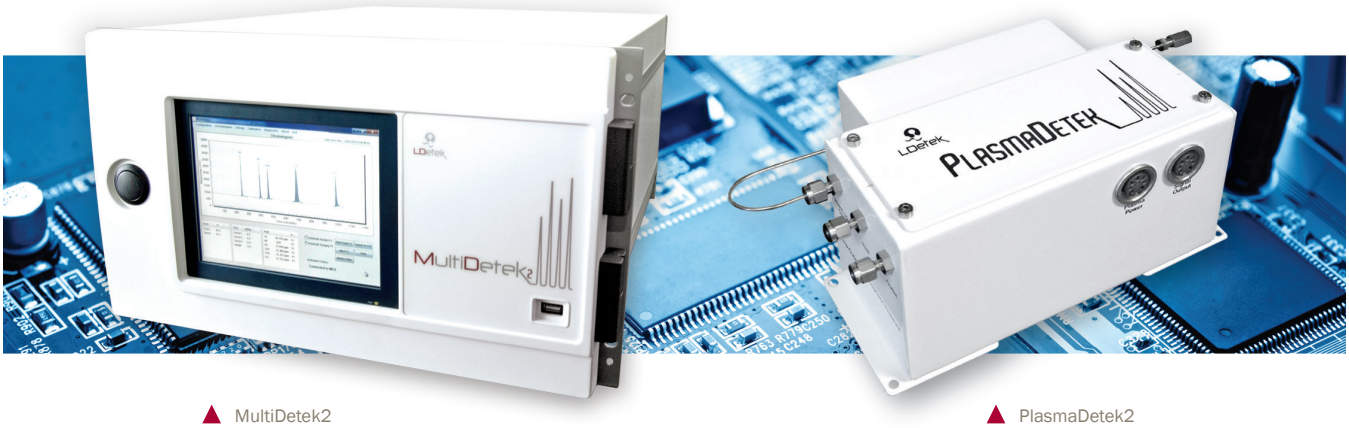
APPLICATION NOTE
LD19-03

Measurement of trace impurities
in high purity nitrous oxide (N20) for electronic gas industry using PlasmaDetek2 and MultiDetek2 GC



▲ MultiDetek2

▲ PlasmaDetek2
patent US 9,310,308 B2

Nitrous oxide (N20), often referred to as laughing gas, is used in the high-tech thin film industries of semiconductor and LCD display manufacturing. The primary application is the reaction with silane (SiH4) or other silicon precursors to produce high-quality oxide films (SiOx), which are used as electrical insulators in microelectronic transistors. N20 is increasingly used to make thin-film oxides with other elements like titanium, aluminium, magnesium and zirconium. It is also used in the selective etching of semiconductor thin-films.

Nitrous oxide is a colourless, non-flammable gas at room temperature with a slightly sweet odour and taste and it is an oxidiser that can support combustion like oxygen. It is an electronic high-purity material produced from thermal decomposition of ammonium nitrate. Why is this gas used in electronics manufacturing? It is less reactive and therefore more selective, than oxygen. Often, this property is used to:

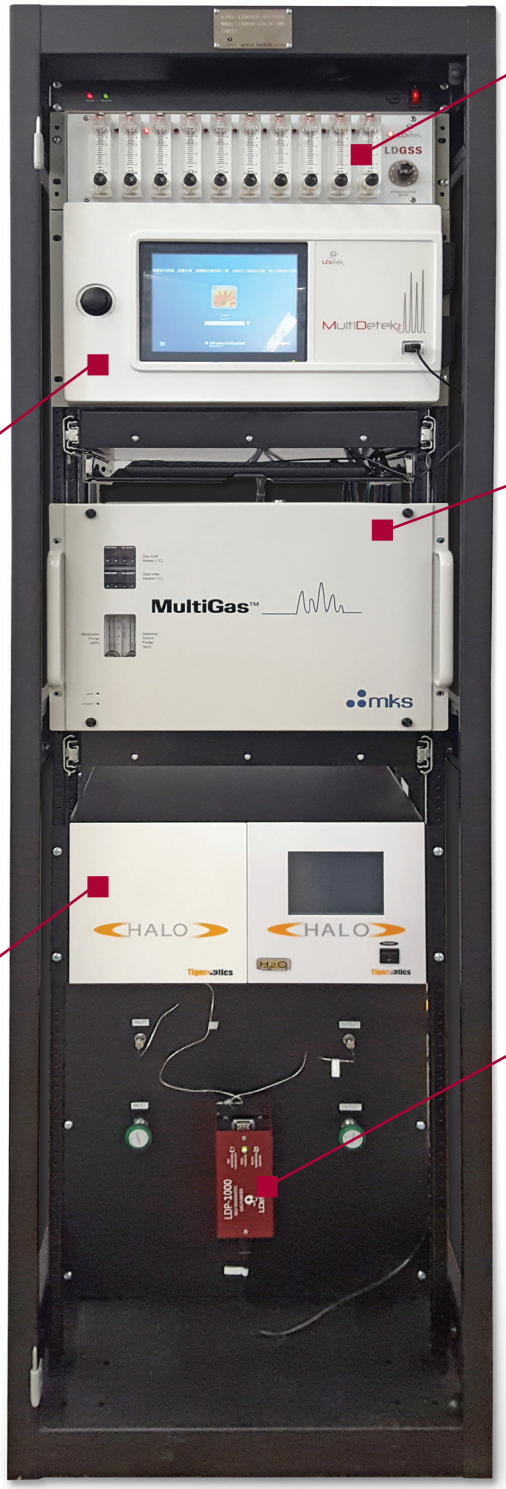
- **Control the amount of oxygen is a thin film**
- **Reduce the side oxidation reactions**
- **Selectively etch one thin film while allowing others to remain**

A addressable market than more than 10,000 metric tons N20 continuously growing with the arrival of new higher-definition display technologies like ultra-high definition and OLED requiring higher amount of N20.

In addition to its use in electronics manufacturing, common applications of nitrous oxide are: anaesthetics, as food and beverage propellant (for whipped cream as example), as an industrial propellant and foaming agent and as a fuel oxidiser for rockets and race cars.

THE COMPLETE SOLUTION

This application note shows the configuration of a complete integrated system (LDrack rackmount cabinet).



LDGSS

ultra high purity gas stream selector using stainless steel diaphragm valves for 10 streams.

MULTIDETEK2

compact/industrial GC for measuring trace ppb/ppm impurities H2-O2-N2-CH4-CO-CO2 with addition of Ar-Ne and addition of C2H2-C2H4-C2H6 all in one instrument using one type of sensor (PlasmaDetek2) with helium as carrier gas.

MKS MULTIGAS

FTIR instrument for measuring ppb/ppm ammonia and NOx.

TIGER OPTICS

Halo instrument for measuring ppb/ppm trace H20.

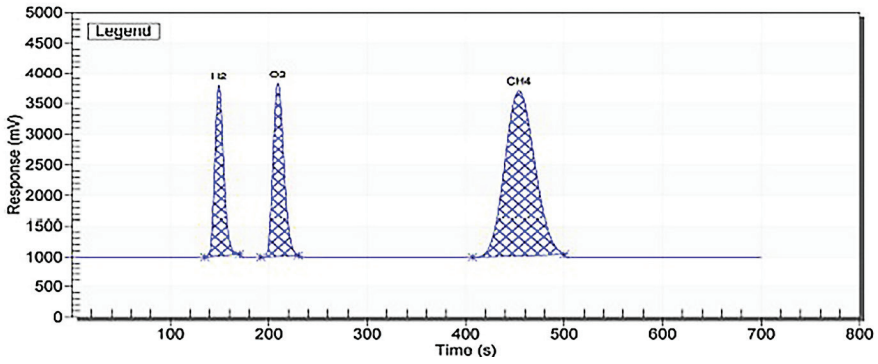
HEATED GAS PURIFIER

for generating grade 99.999999% UHP carrier gas for GC from Helium grade 99.999%

RESULTS:

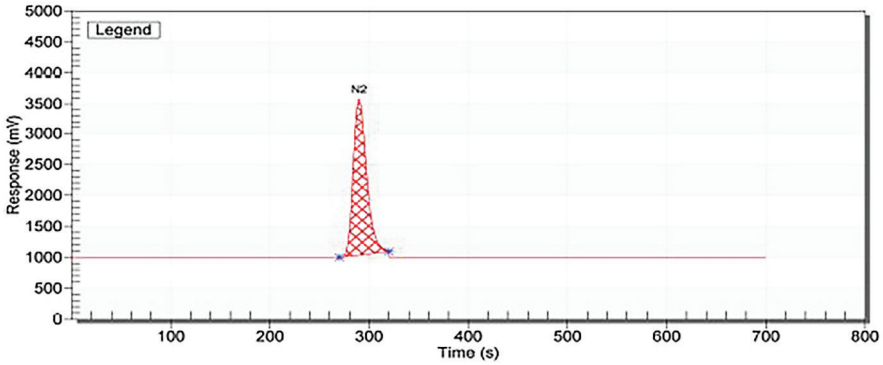
Chromatogram : Trace H2-O2-CH4 impurities in N2O

Peak	Unit	Calibration Value	Area Counts
H2	ppm	5.69	28374
CH4	ppm	5.90	94297
O2	ppm	4.94	35973



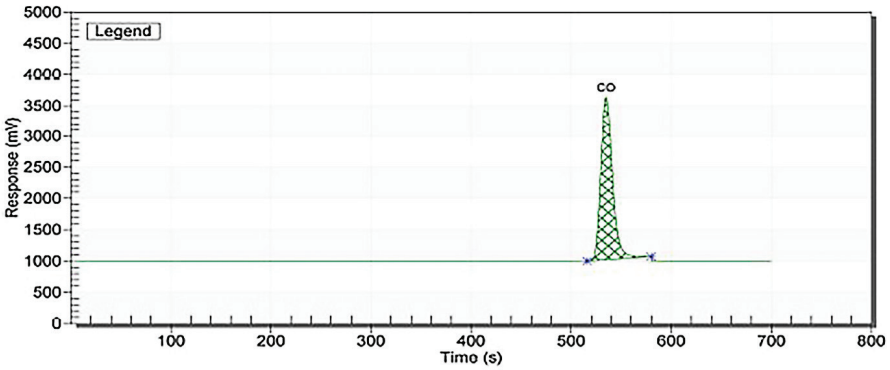
Chromatogram : Trace N2 impurities in N2O

Peak	Unit	Calibration Value	Area Counts
N2	ppm	5.36	39445



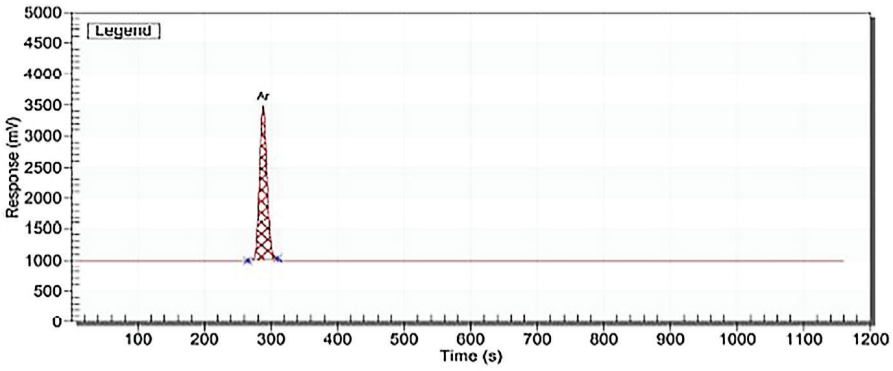
Chromatogram : Trace CO impurities in N2O

Peak	Unit	Calibration Value	Area Counts
CO	ppm	5.32	34990



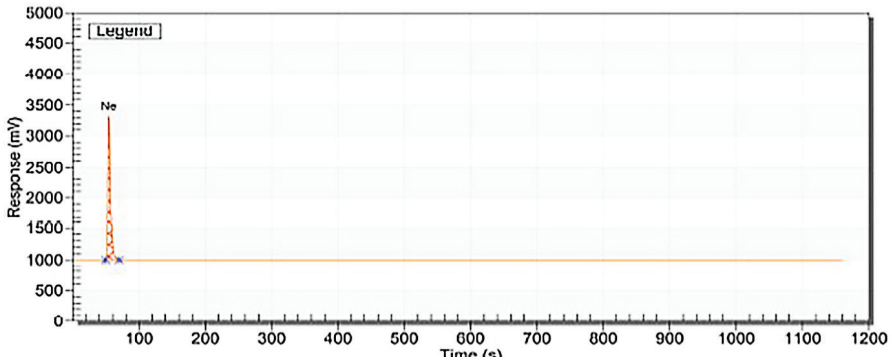
Chromatogram : Trace Ar impurities in N2O

Peak	Unit	Calibration Value	Area Counts
Ar	ppm	4.65	31373



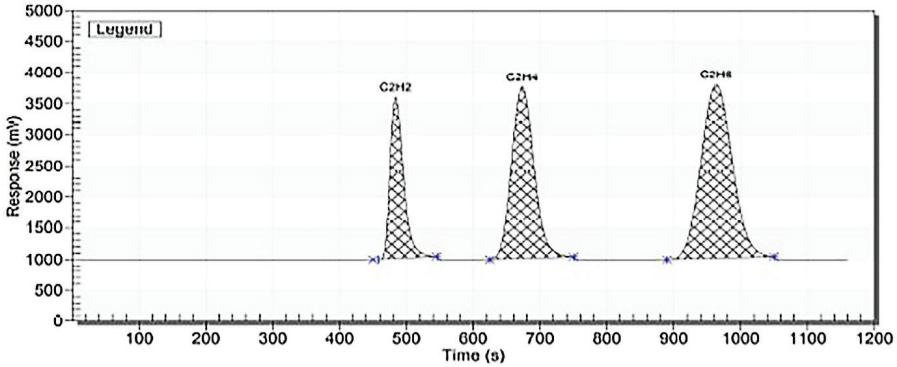
Chromatogram : Trace Ne impurities in N2O

Peak	Unit	Calibration Value	Area Counts
Ne	ppm	4.86	9891



Chromatogram : Trace C2H2-C2H4-C2H6 impurities in N2O

Peak	Unit	Calibration Value	Area Counts
C2H2	ppm	3.72	60461
C2H4	ppm	4.62	107184
C2H6	ppm	4.55	151170



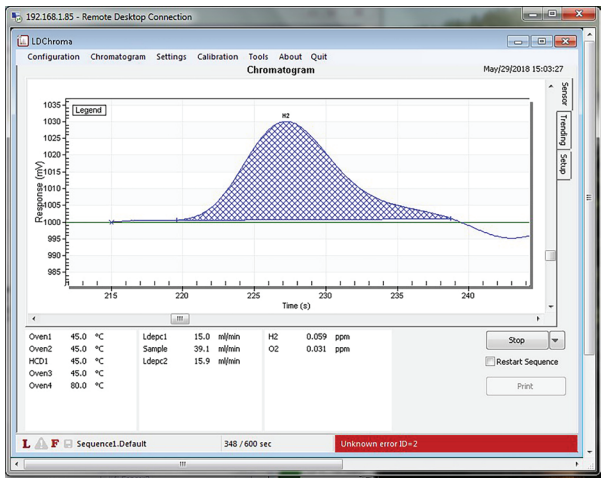
LIMIT OF DETECTION

COMPONENTS	CONCENTRATION	PEAK HEIGHT	NOISE	LDL (3X NOISE)
H2	0.05 ppm	30 mV	2.0 mV	10 ppb
O2	0.053 ppm	30 mV	2.0 mV	10.6 ppb
N2	0.05 ppm	24 mV	1.5 mV	9.3 ppb
CH4	0.05 ppm	33 mV	2.0 mV	9.0 ppb
CO	0.05 ppm	25 mV	2.0 mV	12.0 ppb

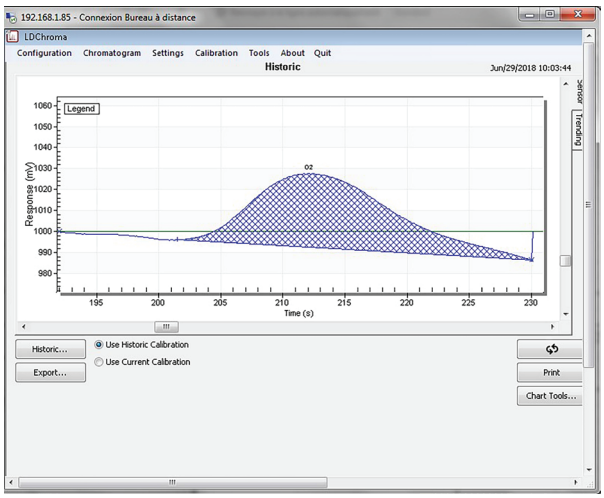
Note: other LDL could be obtained with different injection volume and chromatographic condition

For the Idl of the impurities Ar-Ne-C2H2-C2H4-C2H6, they don't appear in the chart since the results are based on the O2 and CH4 which use the same sensor on the PlasmaDetek2. The Idl is then evaluate at 10ppb for Ar-Ne and 9ppb for C2s.

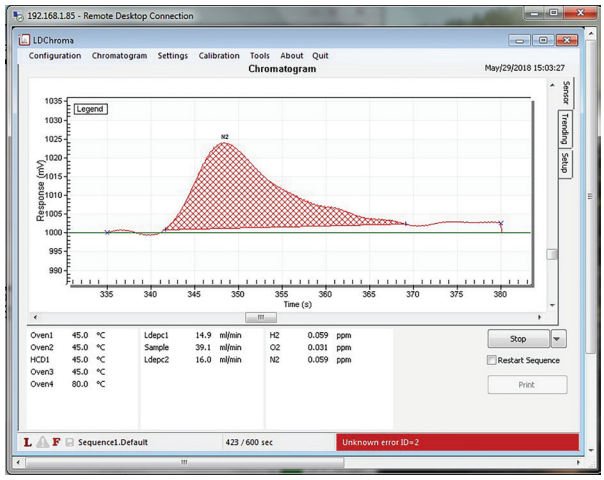
H2 : 0.05ppm



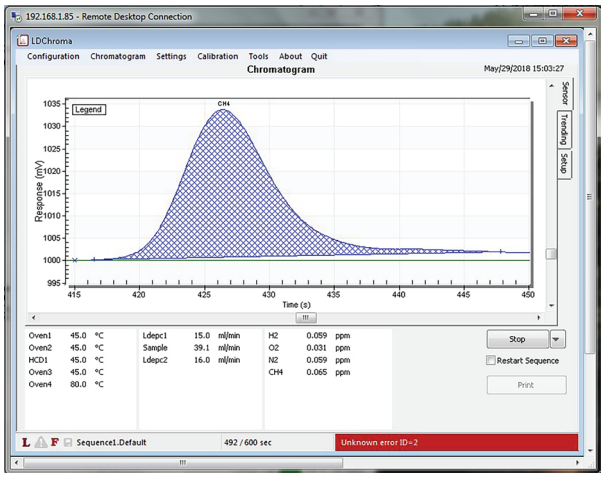
O2 : 0.053ppm



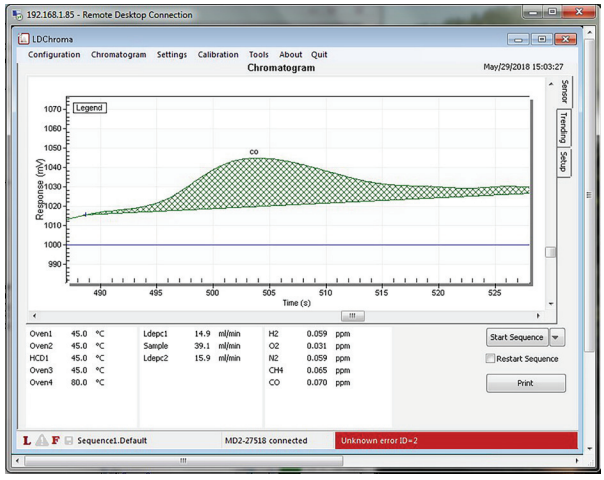
N2 : 0.05ppm



CH4 : 0.05ppm



CO : 0.05ppm

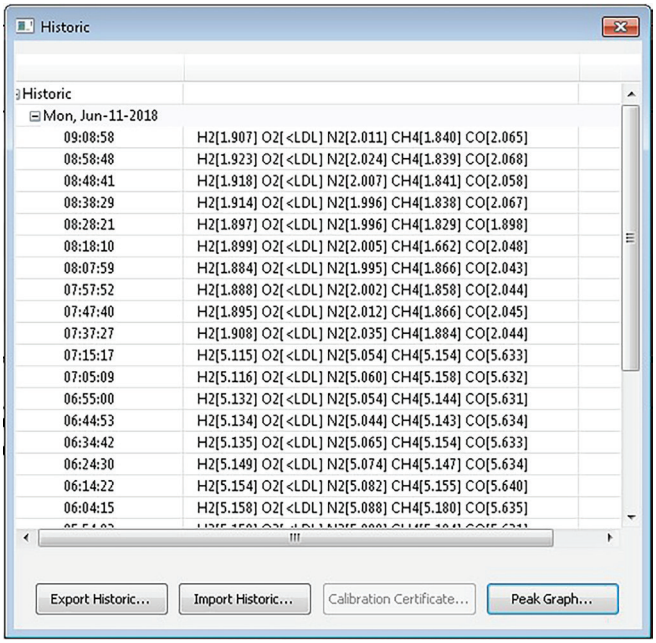


Repeatability to be at a value of CV% x 3 < 5% for a series of consecutive analysis at a fix concentration in a balance gas of nitrous oxide.

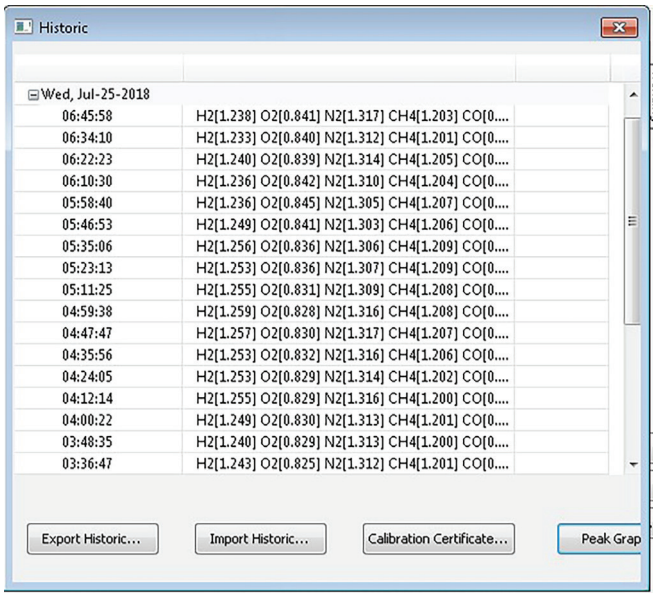
COMPONENTS	Repeatability (CV% x 3)
H2	1.64%
O2	0.73%
N2	1.56%
CH4	2.05%
CO	1.66%

For the repeatability of the impurities Ar-Ne-C2H2-C2H4-C2H6, they don't appear in the chart since the results are based on the O2 and CH4 which use the same sensor on the PlasmaDetek2. An evaluation of 0.73% for Ar-Ne and 2.05% for C2s is given.

Results (screenshot) of consecutive analysis at a fix concentration for H2-N2-CH4-CO impurities in balance N2O



Results (sceenshot) of consecutive analysis at a fix concentration for O2 impurity in balance N2O



CONCLUSION:

Using one PlasmaDetek2 detector inside one unit MultiDetek2 GC, the analysis of trace ppb/ppm impurities H2-O2-N2-CH4-CO-CO2 with addition of Ar-Ne and addition of C2H2-C2H4-C2H6 have been measured. The analysis time for the impurities H2-O2-N2-CH4-CO-CO2 in UHP N2O was realized within 13 minutes. By adding the analysis of Ar-N2-C2H2-C2H4-C2H6 on top of the other impurities listed, the analysis time was realized in 20 minutes. The MultiDetek2 GC was configured with helium as carrier gas, stainless steel diaphragm valve and capillary MXT type columns to minimize the carrier flow consumption. The unit is using 4-20mA outputs for each impurity and also the Modbus protocol for transmitting the data's. The GC and the stream selector system LDGSS are both remotely controlled by the Ethernet port. Meaning that the calibration of the units can be performed remotely.

The complete integration of the system has been made inside a standard rackmount enclosure. The instruments from Tiger Optics and from MKS have been added for the analysis of H2O and NOx-NH3 respectively.

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