APPLICATION NOTE
LD18-02

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

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 enclosure. The analyses of trace sub ppb impurities of H2-Ar-N2-CH4-CO-CO2-NMHC can be performed in multiple gas backgrounds. This application note will show the results obtained for sub ppb trace H2-CO-N2-CH4-CO2 in Argon-Helium-Oxygen.

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 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 and/or FID as commonly installed by other GC manufacturers.

This document demonstrates the performances of the system by offering chromatograms all obtained at low ppb concentration to show the real peak shapes and results in an industrial environment. For more details about other UHP gases application notes for SEMI , please refer to the application note LD15-02 and LD16-03 that give 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

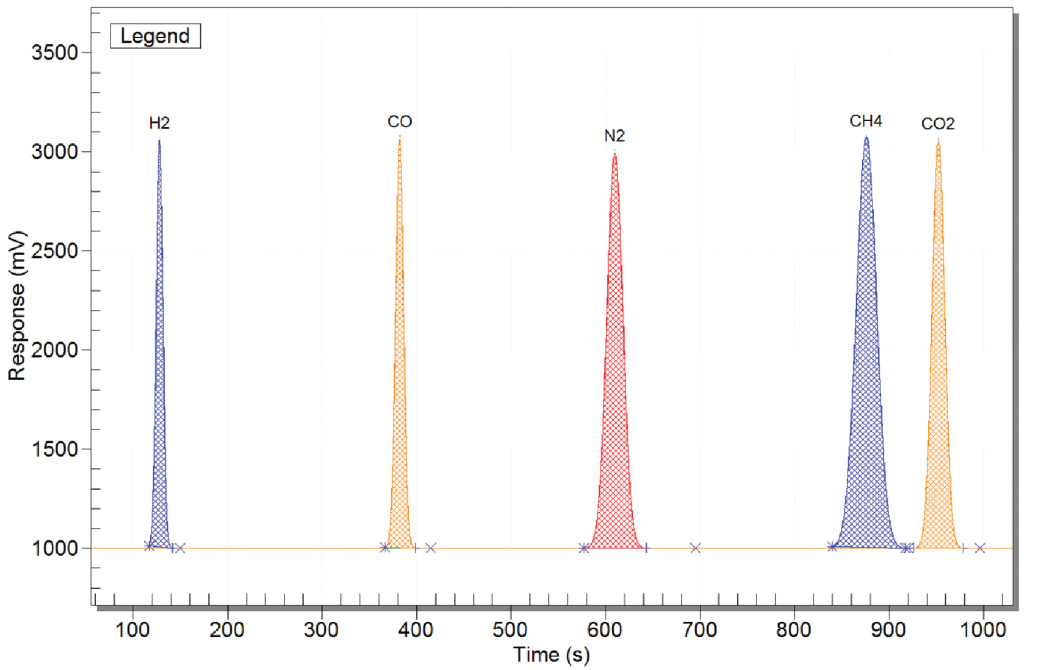
Chart 1: MultiDetek2 multiple configuration capabilities

RESULTS:

The results demonstrated have been performed in a real industrial environment. The chromatograms and results have been obtained by measuring different concentrations between 0ppb and 100ppb for the different impurities. It shows and defines the stability, the accuracy, and the LDL.

Our tests have been performed using our LDGDS gas dilution system to generate real low ppb concentrations. Our LDL results are based on real dilutions and not based on a signal to noise ratio.

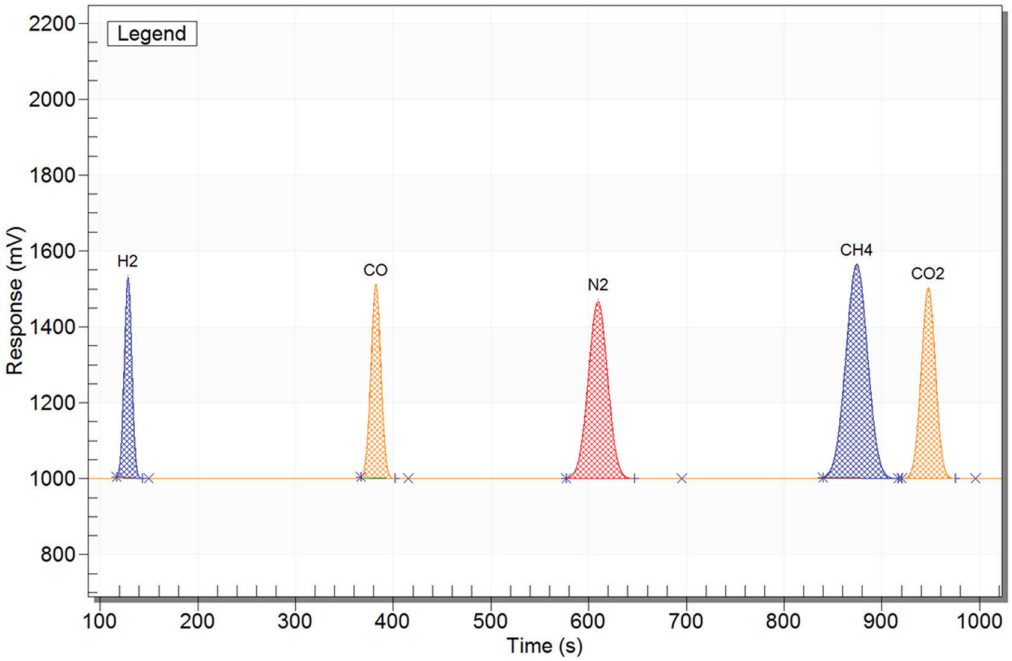
CHROMATOGRAM AT 100PPB



STABILITY/ACCURACY CHART AT 100PPB

100ppb	H2	CO	N2	CH4	CO2
sam, 24 mars 2018 09:19:01	100.057	99.980	100.157	100.135	100.439
sam, 24 mars 2018 09:35:15	100.165	99.960	100.169	100.074	100.400
sam, 24 mars 2018 09:53:39	100.132	100.056	100.199	100.079	100.517
sam, 24 mars 2018 10:12:03	100.110	100.164	100.219	100.111	100.500
sam, 24 mars 2018 10:28:01	100.094	100.116	100.239	100.114	100.424
sam, 24 mars 2018 10:46:25	100.116	100.069	100.330	100.217	100.361
sam, 24 mars 2018 11:04:49	100.209	100.060	100.381	100.275	100.471
sam, 24 mars 2018 11:23:14	100.276	100.012	100.217	100.296	100.488
sam, 24 mars 2018 11:39:14	100.250	100.051	100.275	100.345	100.546
sam, 24 mars 2018 11:57:36	100.236	100.127	100.252	100.320	100.589
min	100.057	99.960	100.157	100.074	100.361
max	100.276	100.164	100.381	100.345	100.589
diff	0.219	0.204	0.224	0.271	0.228
avg	100.165	100.060	100.244	100.197	100.474

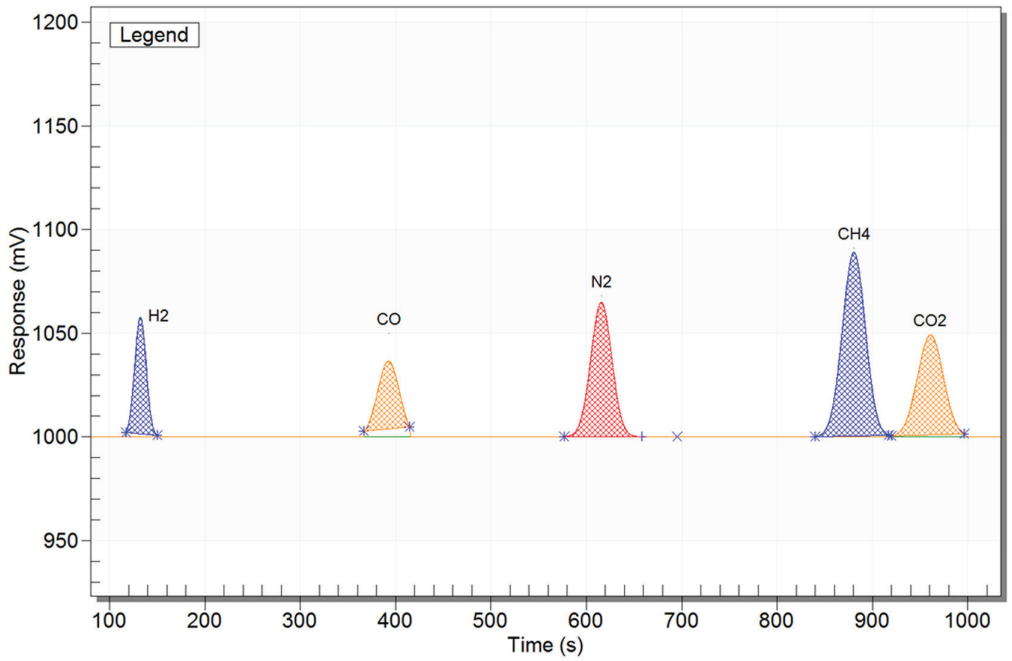
CHROMATOGRAM AT 25PPB



STABILITY/ACCURACY CHART AT 25PPB

25ppb	H2	CO	N2	CH4	CO2
lun, 19 mars 2018 19:33:30	26.431	28.258	23.472	25.897	24.918
lun, 19 mars 2018 19:49:30	26.427	28.350	23.295	25.889	24.966
lun, 19 mars 2018 20:07:53	26.349	28.178	23.403	26.019	25.157
lun, 19 mars 2018 21:41:40	26.511	28.144	23.519	26.096	24.971
lun, 19 mars 2018 21:57:36	26.520	28.324	23.457	25.844	25.030
lun, 19 mars 2018 22:15:59	26.665	28.475	23.351	25.974	25.074
lun, 19 mars 2018 22:34:21	26.521	28.361	23.383	26.153	25.042
lun, 19 mars 2018 22:50:29	26.520	28.431	23.363	25.981	24.959
lun, 19 mars 2018 23:08:54	26.539	28.457	23.369	26.003	25.074
lun, 19 mars 2018 23:27:17	26.477	28.155	23.319	26.037	25.215
min	26.349	28.144	23.295	25.844	24.918
max	26.665	28.475	23.519	26.153	25.215
diff	0.316	0.331	0.224	0.309	0.297
avg	26.496	28.313	23.393	25.989	25.041

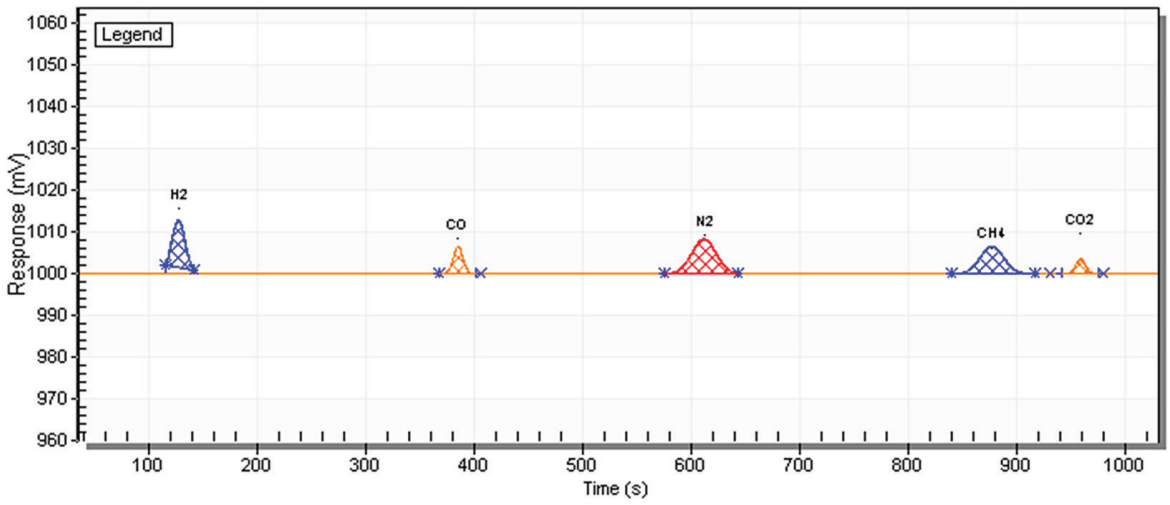
CHROMATOGRAM AT 4PPB



STABILITY/ACCURACY CHART AT 4PPB

4ppb	H2	CO	N2	CH4	CO2
dim, 25 mars 2018 12:53:16	4.393	3.870	3.869	4.567	4.359
dim, 25 mars 2018 13:11:40	4.377	3.918	3.869	4.561	4.383
dim, 25 mars 2018 13:27:42	4.402	3.921	3.864	4.589	4.321
dim, 25 mars 2018 13:46:05	4.389	4.070	3.861	4.593	4.283
dim, 25 mars 2018 14:04:29	4.396	3.915	3.860	4.617	4.395
dim, 25 mars 2018 14:20:29	4.409	3.879	3.864	4.617	4.323
dim, 25 mars 2018 14:38:53	4.397	3.935	3.857	4.634	4.243
dim, 25 mars 2018 14:57:19	4.388	3.978	3.865	4.620	4.443
dim, 25 mars 2018 15:13:18	4.379	4.091	3.865	4.638	4.421
dim, 25 mars 2018 15:31:18	4.386	3.900	3.867	4.683	4.255
min	4.377	3.870	3.857	4.561	4.243
max	4.409	4.091	3.869	4.683	4.443
diff	0.032	0.221	0.012	0.122	0.200
avg	4.392	3.948	3.864	4.612	4.343

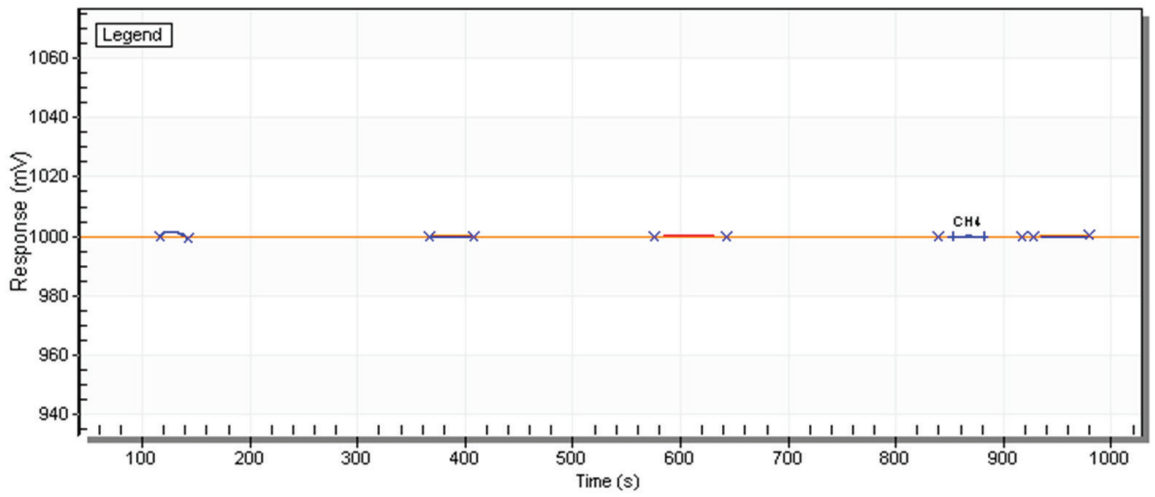
CHROMATOGRAM AT 1PPB



STABILITY/ACCURACY CHART AT 1PPB

1 ppb	H2	CO	N2	CH4	CO2
mer, 28 mars 2018 08:06:07	1,004	0,896	1,085	1,048	1,067
mer, 28 mars 2018 08:27:42	0,983	0,888	1,011	1,046	1,062
mer, 28 mars 2018 08:49:04	0,971	0,875	1,016	1,048	1,065
mer, 28 mars 2018 09:10:17	0,970	0,891	1,100	1,045	1,067
mer, 28 mars 2018 09:28:38	0,962	0,896	1,098	1,047	1,063
mer, 28 mars 2018 09:46:42	0,982	0,895	1,050	1,037	1,065
mer, 28 mars 2018 10:04:57	0,980	0,885	1,052	1,043	1,063
min	0,962	0,875	1,011	1,037	1,062
max	1,004	0,896	1,100	1,048	1,067
diff	0,042	0,021	0,089	0,011	0,005
avg	0,979	0,889	1,059	1,045	1,065

CHROMATOGRAM WITH UHP PURIFIED HELIUM

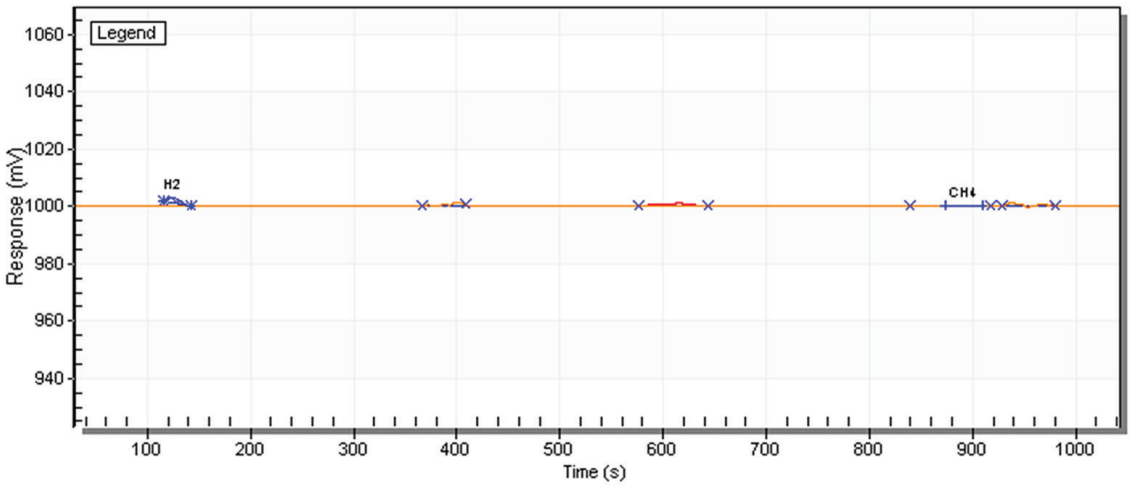


STABILITY/ACCURACY CHART WITH UHP PURIFIED HELIUM

Pure Helium	H2	CO	N2	CH4	CO2
mar, 27 mars 2018 18:42:26	<LDL	<LDL	<LDL	<LDL	<LDL
mar, 27 mars 2018 19:00:49	<LDL	<LDL	<LDL	<LDL	<LDL
mar, 27 mars 2018 19:16:51	<LDL	<LDL	<LDL	<LDL	<LDL
mar, 27 mars 2018 19:35:14	<LDL	<LDL	<LDL	<LDL	<LDL
mar, 27 mars 2018 19:53:37	<LDL	<LDL	<LDL	<LDL	<LDL
mar, 27 mars 2018 20:09:43	<LDL	<LDL	<LDL	<LDL	<LDL
mar, 27 mars 2018 20:28:05	<LDL	<LDL	<LDL	<LDL	<LDL
min	0,000	0,000	0,000	0,000	0,000
max	0,000	0,000	0,000	0,000	0,000
diff	0,000	0,000	0,000	0,000	0,000
avg	0,000	0,000	0,000	0,000	0,000

LDL value set at 0.5ppb

CHROMATOGRAM WITH UHP PURIFIED ARGON



STABILITY/ACCURACY CHART WITH UHP PURIFIED ARGON

Pure Argon	H2	CO	N2	CH4	CO2
lun, 26 mars 2018 18:32:46	<LDL	<LDL	<LDL	<LDL	<LDL
lun, 26 mars 2018 18:51:08	<LDL	<LDL	<LDL	<LDL	<LDL
lun, 26 mars 2018 19:07:09	<LDL	<LDL	<LDL	<LDL	<LDL
lun, 26 mars 2018 19:25:32	<LDL	<LDL	<LDL	<LDL	<LDL
lun, 26 mars 2018 19:43:57	<LDL	<LDL	<LDL	<LDL	<LDL
lun, 26 mars 2018 20:02:19	<LDL	<LDL	<LDL	<LDL	<LDL
lun, 26 mars 2018 20:18:21	<LDL	<LDL	<LDL	<LDL	<LDL
min	0,000	0,000	0,000	0,000	0,000
max	0,000	0,000	0,000	0,000	0,000
diff	0,000	0,000	0,000	0,000	0,000
avg	0,000	0,000	0,000	0,000	0,000

LDL value set at 0.5ppb

CONCLUSION:

Our complete cabinet integration with our MultiDetek2 (GC), PlasmaDetek2 (PED), LDGSS gas stream selector, LDGDS gas dilution system and our LDP1000 carrier gas purifier are all perfectly designed and approved for the UHP semiconductor applications. With the results demonstrated, we can clearly see our systems is capable of generating and detecting very clear peaks at sub ppb concentrations. Don't hesitate to contact LDetek if more informations are required.

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