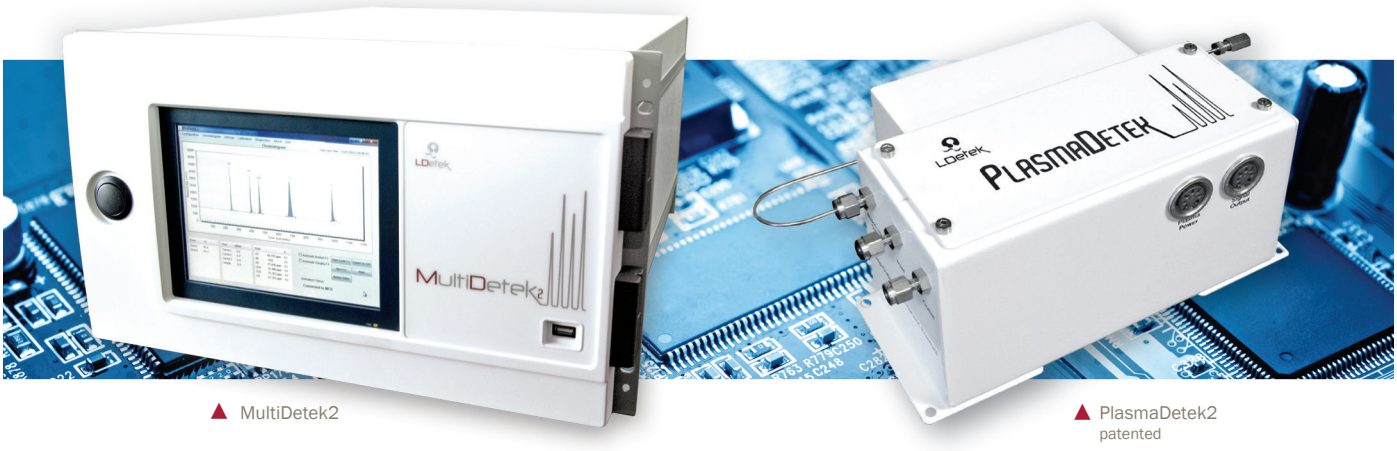
APPLICATION NOTE

LD16-03

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



▲ MultiDetek2

▲ PlasmaDetek2
patented

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

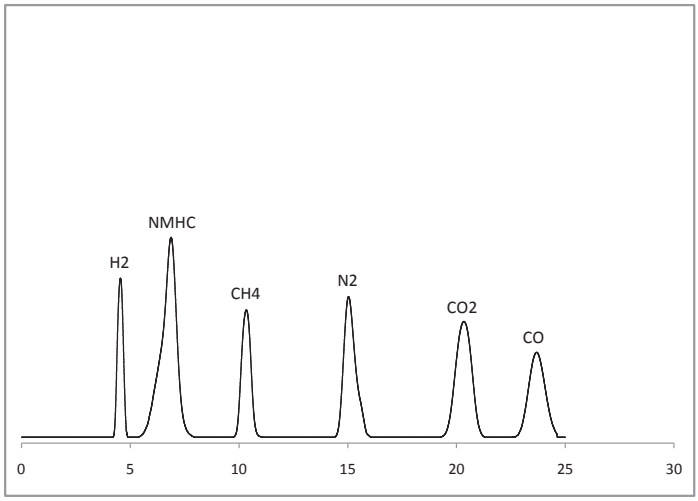
RESULTS:

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)

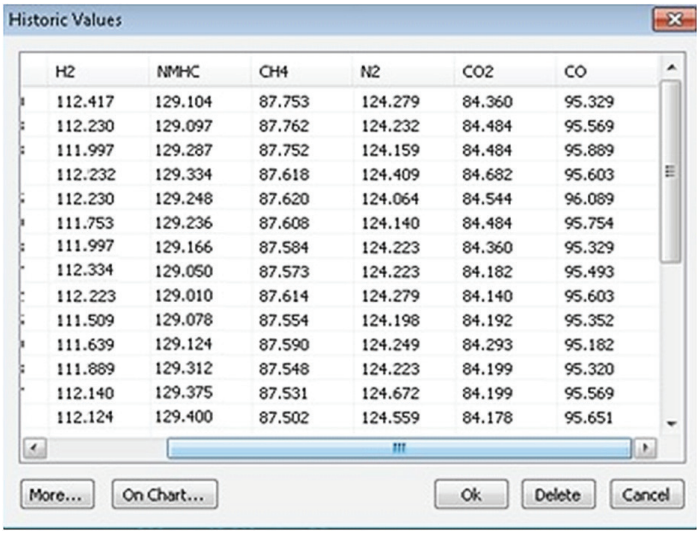
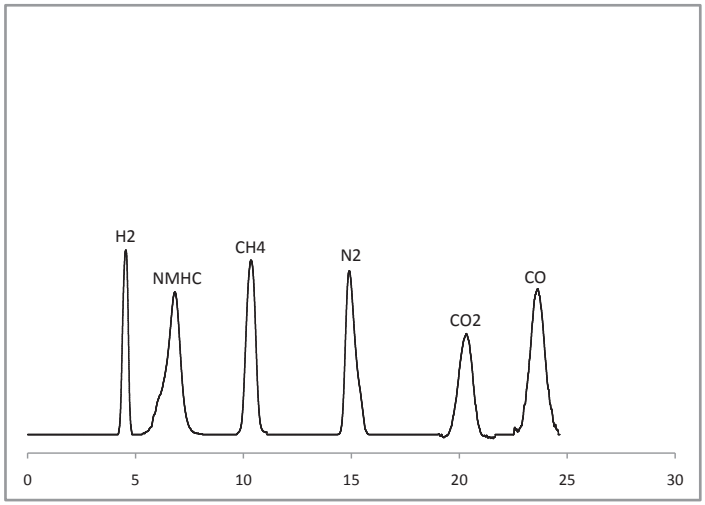


Figure 1 : Results of ten consecutive cycles used for accuracy and stability calculation



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)

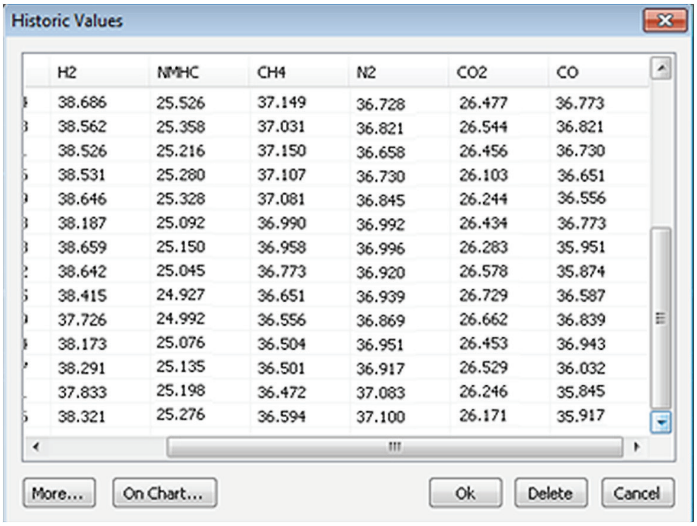
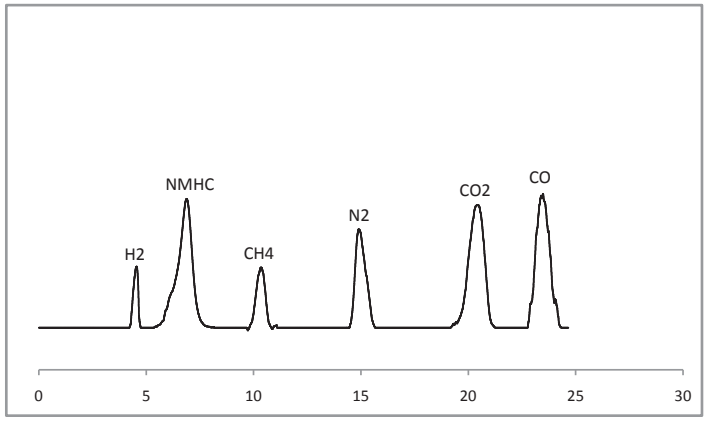


Figure 2 :
Results of ten consecutive cycles
used for accuracy and stability calculation



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)

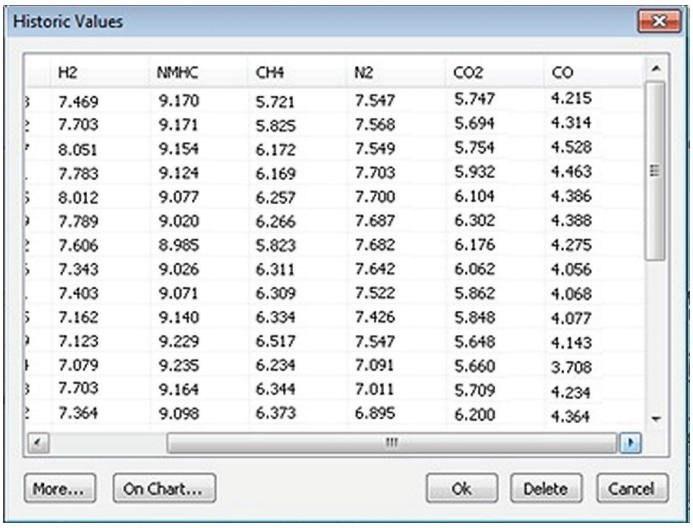
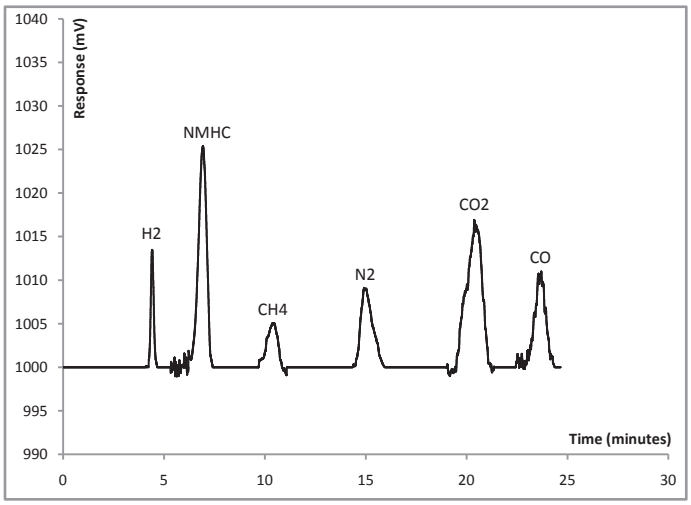


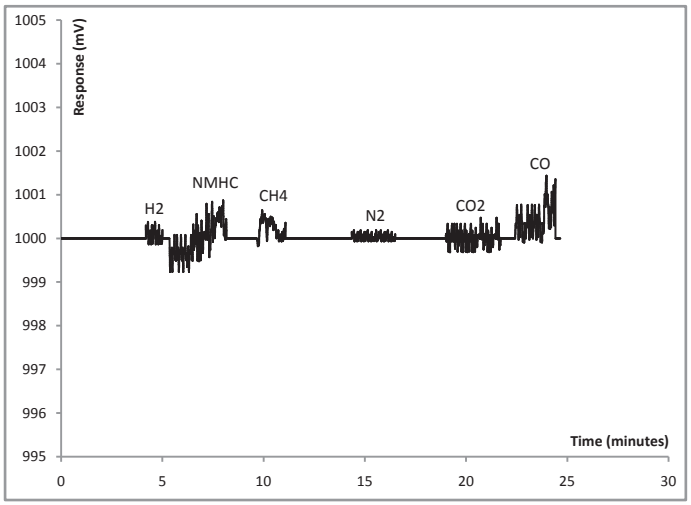
Figure 3 :
Results of ten consecutive cycles used for
accuracy and stability calculation

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.



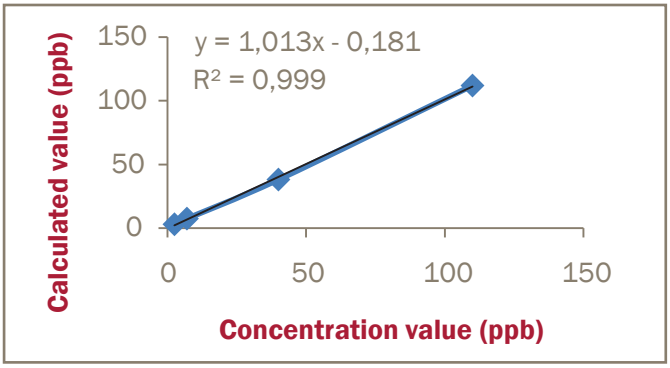
Chromatogram 5:
Baseline noise analysis used for LDL calculation

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

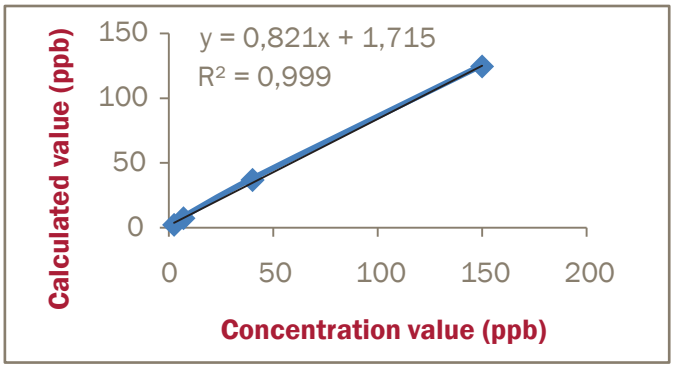
Chart 5: LDL calculation (results are dependent on the system conditions and can vary)

LINEARITY :

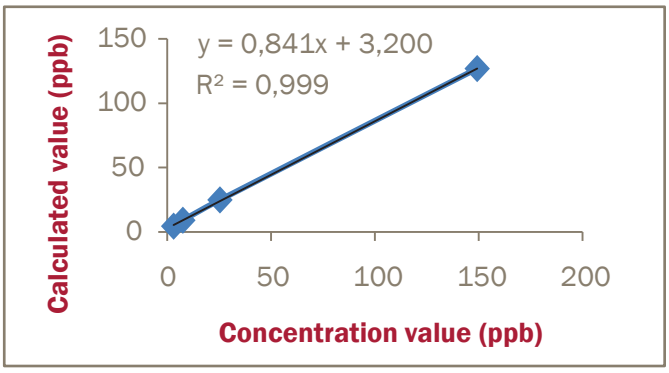
The linearity is calculated by running a minimum of 10 cycles at four different concentrations between 0 and 500ppb. The average of the results of the 10 cycles is used to generate the calculated values which are compared to a linear trend in relation to the known concentration values. The graphs 1-2-3-4-5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



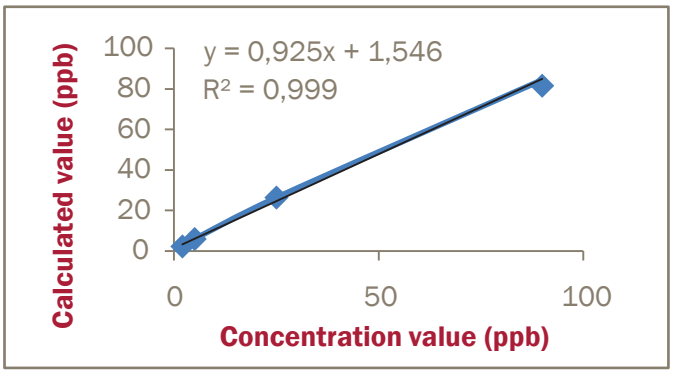
Graph 1: H_2 peak linearity



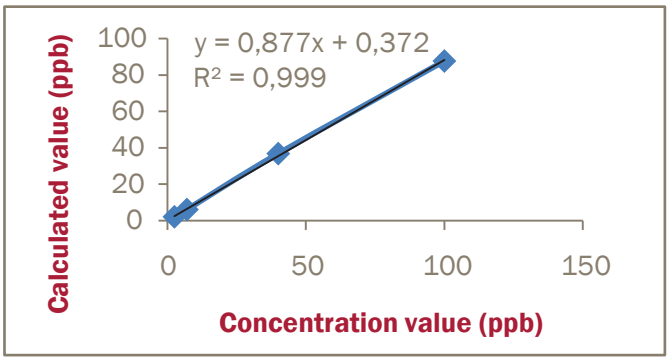
Graph 4: N_2 peak linearity



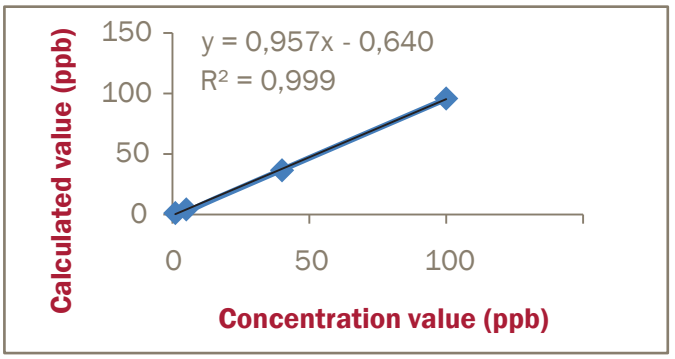
Graph 2: NMHC peak linearity



Graph 5: CO_2 peak linearity



Graph 3: CH_4 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..

LDetek

Where innovation leads to success

271 St-Alphonse Sud, Thetford Mines, Quebec, Canada, G6G 3V7

Phone: 418 755-1319 • Fax: 418 755-1329 • **info@ldetek.com** • **www.ldetek.com**