

APPLICATION NOTE

LD18-01



Measurement of trace impurities in Germane (GeH₄) for electronic gas industry using PlasmaDetek2 and MultiDetek2



▲ MultiDetek2

▲ PlasmaDetek2
patent US 9,310,308 B2

LDETEK SOLUTION:

Handling highly flammable gas like germane (GeH₄) requires a high level of safety and it 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 purged box is fully welded offering no possibility of leakage.

Step 2: A selection valve is mounted before the diaphragm valves used for filling the sampling loops in the system. That selection valve is configured to allow GeH₄ going to the sampling loops only for a pre determined period of time just before to start each analysis. The short period of time is configured in factory and is dependant to the system configuration. The rest of the time, the selection valve is switched on a purged gas which is normally the same gas type that 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 GeH₄ 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 and the diaphragm selection valve is mounted in the MultiDetek2 chassis. That box is normally purged with the same gas type that the carrier gas used for the system. That environment requires low purge flow rate of about 10-30sccm through a fix orifice depending of the system configuration. The box is fully ambient air free and the system is ready to use after a short period of about 20-30minutes depending of the system configuration. This waiting period is only requires 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 GeH4. 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 potential presence of GeH4. If trace of air is measured in the purged box by the micro PED, then an alarm is activated to shut off the flow of GeH4 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 requires 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 is controlled with the electronic flow controller to ensure a stable and constant flow rate whichever the gas type selected.

The GC configuration for this application is configured with Stainless Steel purged diaphragm valves and MXT column types. At the end of the GC configuration, the PlasmaDetek-2 detector is configured to selectively measure the impurities requested. Multiple channels can be configured depending of the application. For this application note, the MultiDetek2 GC is configured as follow:

Trace impurities C2H2-C2H4-C2H6 → is measured through channel #1

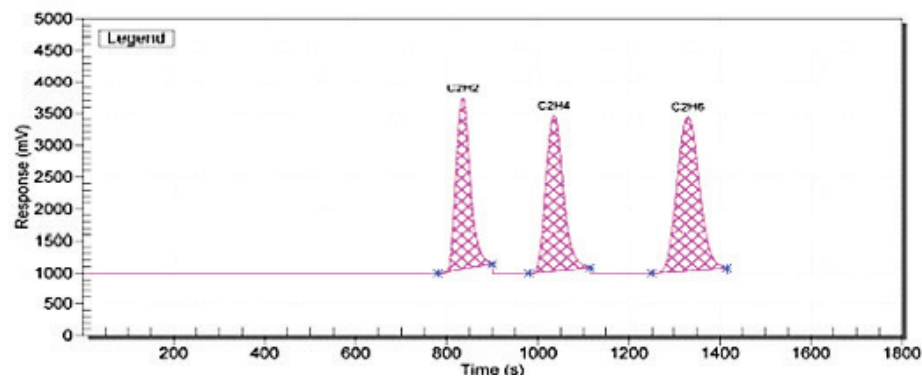
Trace impurities Ar → is measured through channel #2

Trace impurities CO-CO2 → is measured through channel #3

Trace impurities H2-O2-N2-CH4 → is measured through channel #4

RESULTS:

Chromatograms : Trace impurities in GeH4



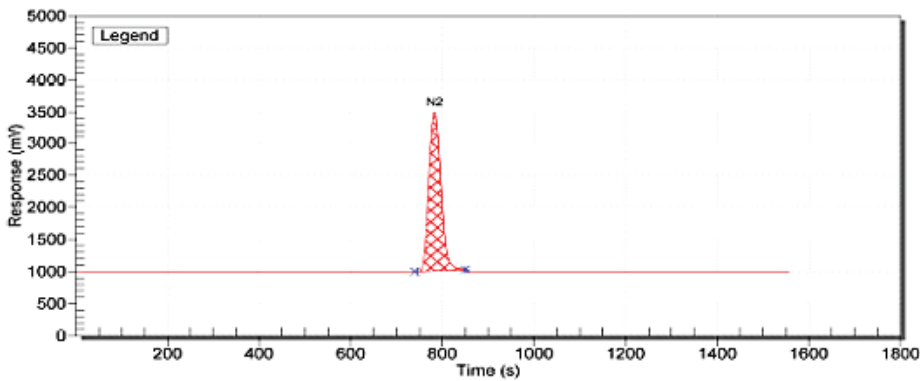
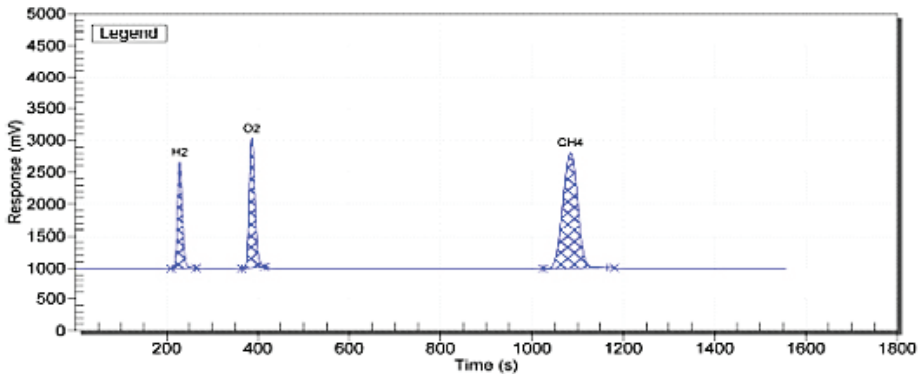
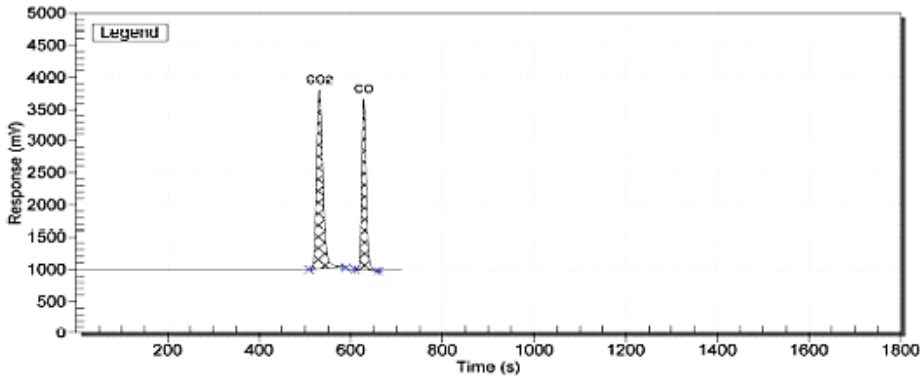
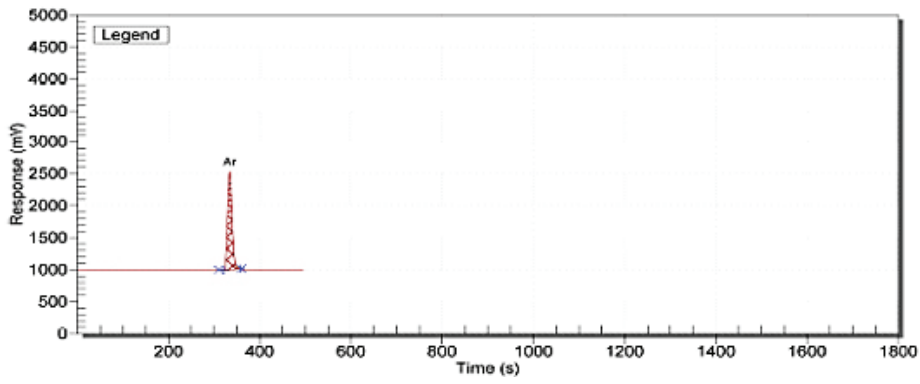


CHART: RESULTS, RESPONSE AND LDL CALCULATION

COMPONENTS	CONCENTRATION	PEAK HEIGHT	NOISE	LDL (3X NOISE)
C2H2	5.2 ppm	2720 mV	2.8 mV	0.016 ppm
C2H4	4.9 ppm	2495 mV	2.8 mV	0.016 ppm
C2H6	4.9 ppm	2433 mV	2.8 mV	0.017 ppm
Ar	3.1 ppm	1544 mV	1.1 mV	0.006 ppm
CO2	4.7 ppm	2802 mV	2.1 mV	0.010 ppm
CO	4.6 ppm	2705 mV	2.1 mV	0.010 ppm
H2	3.9 ppm	1701 mV	2.6 mV	0.018 ppm
O2	4.1 ppm	2065 mV	2.6 mV	0.015 ppm
CH4	3.6 ppm	1789 mV	2.6 mV	0.016 ppm
N2	3.7 ppm	2505 mV	0.7 mV	0.003 ppm

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

CONCLUSION:

The PlasmaDetek2 and the MultiDetek2 combined with the highly safe continuous monitoring sampling system allows the measurement of GeH4 purity with reduced risk. The N2 monitoring of the purged box is essential to ensure there is no ignition risk inside the MultiDetek2.

The trace impurities measurement can be realized with a relatively short analysis time and can offer very low limit of detection of the measured impurities what is required for GeH4 purity analysis.



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