

# APPLICATION NOTE

# LD26-02



## Analysis of trace impurities in UHP Germane



Germane is a highly toxic, flammable, and colorless gas primarily used for the solar panel, the semiconductor and the electronics industries for specialized applications. It is a hydride of germanium, often referred to as germanium tetrahydride, that serves as a vital source of germanium in chemical processes.

### KEY UTILITY AND APPLICATIONS

- **Solar Panels:** It is used in the production of high-efficiency photovoltaic cells (solar panels) to improve energy conversion efficiency.
- **Semiconductor Manufacturing:** Germane is used in the epitaxial growth of silicon-germanium (SiGe) alloy layers to produce high-performance, high-speed integrated circuits and devices.
- **Chemical Vapor Deposition (CVD):** It is a key precursor in CVD and molecular beam epitaxy (MBE) processes, which allow for the creation of thin films, specifically germanium carbide semiconducting films.
- **Optical Fibers:** Germane is utilized in the production of specialty glasses and optical fibers.
- **Material Preparation:** Used for the preparation of high-purity Germanium (Ge) metal.

## LDETEK SOLUTION

The purity of germane can be qualified with the use of the MultiDetek3 gas chromatograph configured with PED.

The unit is configured with a measuring range of 0-10ppm and Idl of 5-10ppb for impurities (Ar+O2)-N2-CH4-CO-C2H6-C3H8-C4H10-CO2 in a sample gas UHP germane(GeH4). The PED (plasma emission detector) has been mounted in the GC to measure the ppb impurities in UHP germane using Helium as carrier gas. All previously listed impurities are measured within one single analyser.

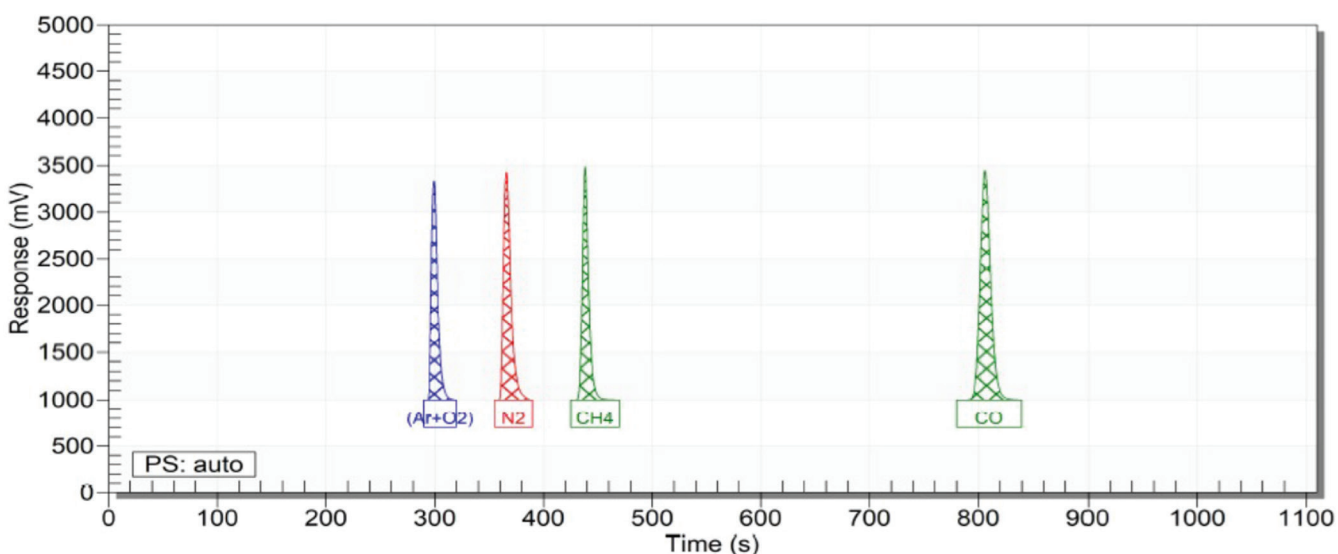
As the germane is highly flammable, pyrophoric and toxic, our GC configuration offers here a specific configuration of the sample flow path. A multiport selection valve mounted in a purged box which is purged with the helium carrier gas ensures the sample is not in contact with its surrounding air. The selection valve ensures to minimize the period which the silane is going into the injection's valves and the sample loops. A specific sequence is configured to have mostly helium in the GC sample flow path with a small period having the silane filling the sampling loops for proper analysis. The sequence is built in consequence to properly purged the loops prior to fill with sample gas germane. This solution offers the GC to have an improved safety level and have an extended lifetime due to the limited time of the components in contact with GeH4.

The crude product germane where analysis of % GeH4 in balance gas H2 can be measured with a TCD with the proper columns and valves configuration channel. See the 5% GeH4 in balance gas hydrogen below.

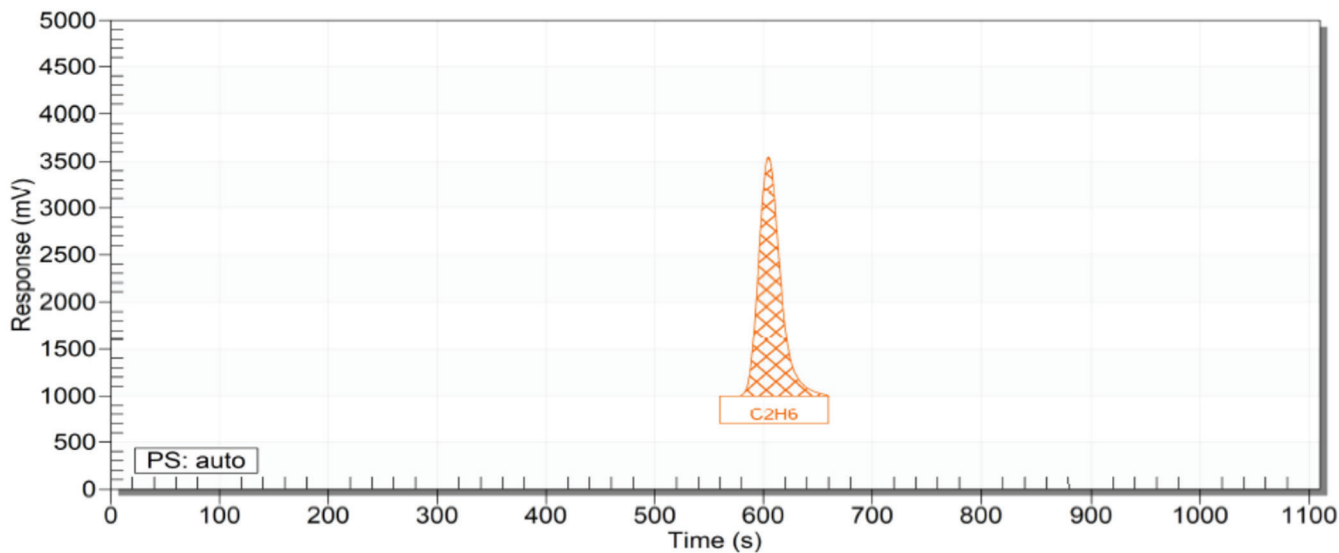
Other configurations and ranges/Idls are possible. The parameters mostly depend on the site production requirements and process.

## RESULTS

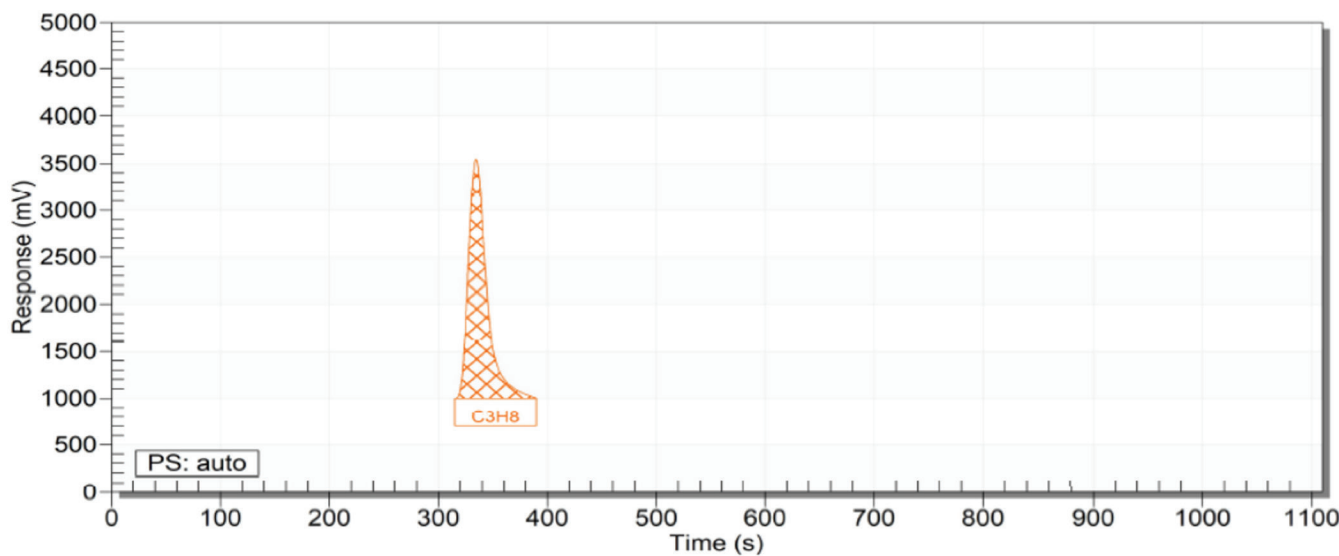
A series of chromatograms (Span calibration) of trace impurities (Ar+O2)-N2-CH4-CO-C2H6-C3H8-C4H10-CO2 in balance gas UHP Germane (GeH4)



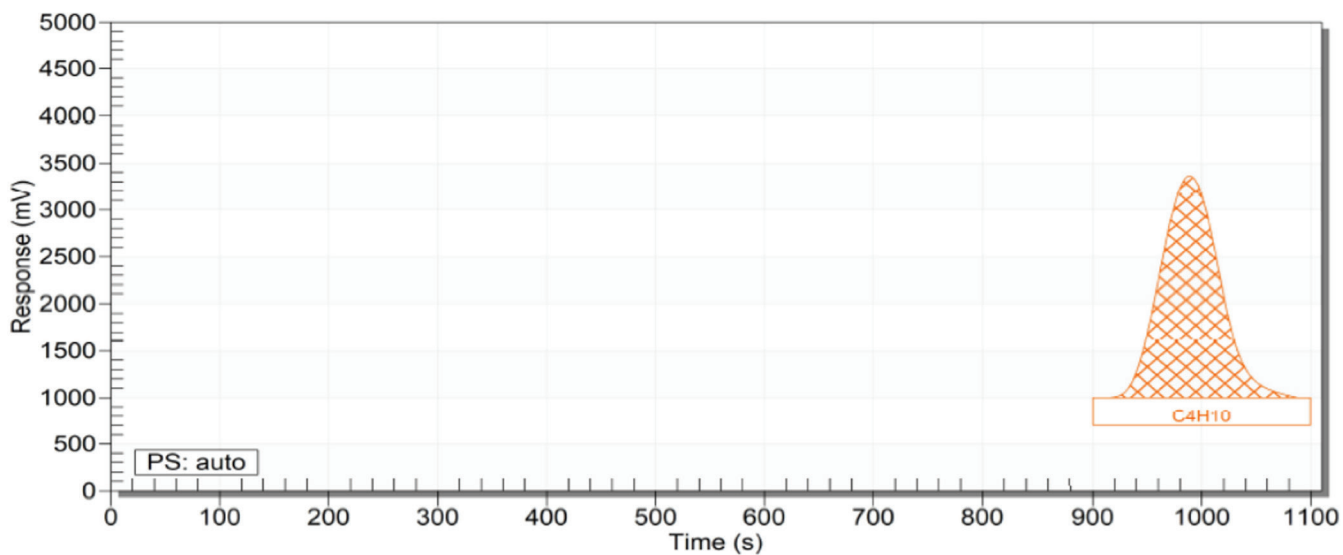
Peak	Unit	Calibration Value	_Area Counts
(Ar+O2)	ppm	9.32	2613
N2	ppm	9.69	17115
CH4	ppm	10.00	22257
CO	ppm	9.85	30671



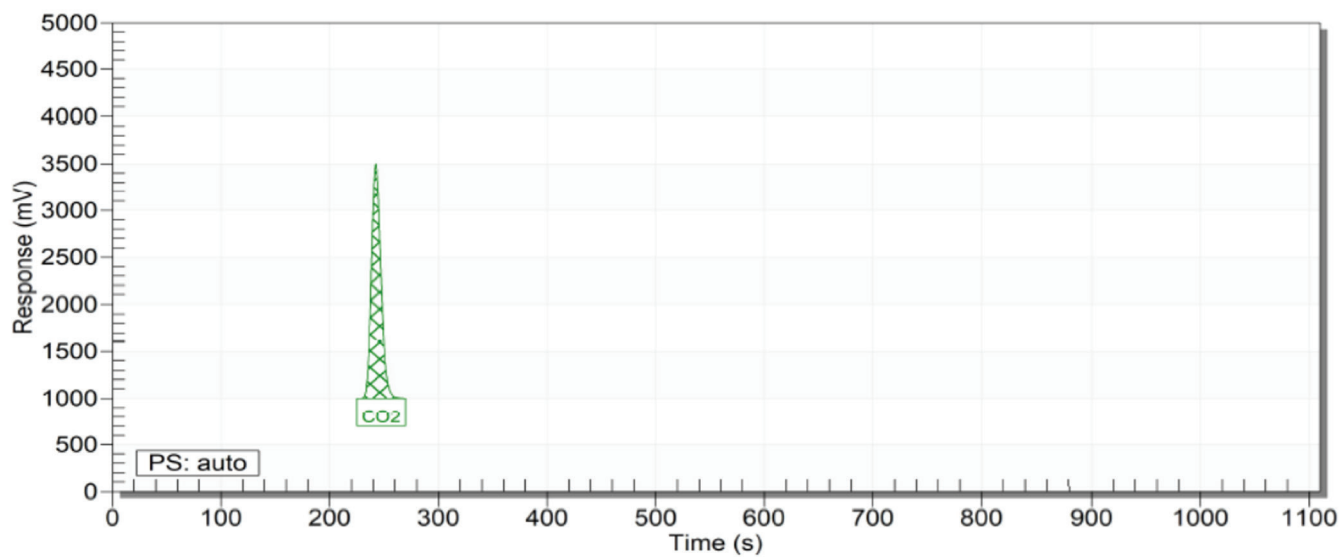
Peak	Unit	Calibration Value	_Area Counts
C2H6	ppm	10.00	13879



Peak	Unit	Calibration Value	_Area Counts
C3H8	ppm	10.00	17795

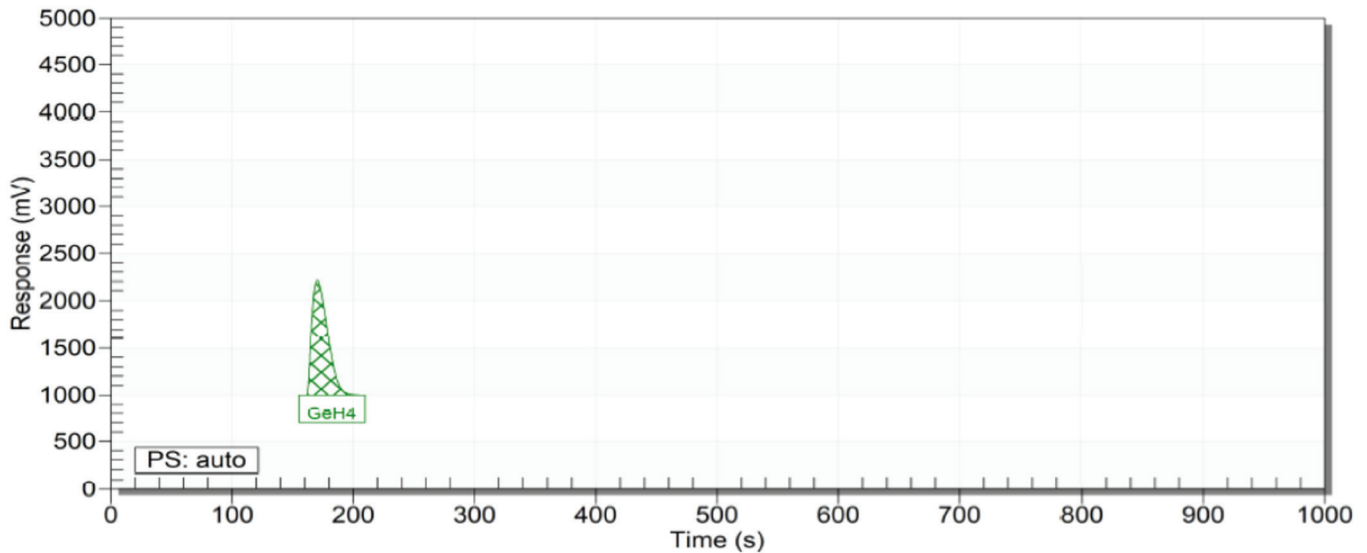


Peak	Unit	Calibration Value	_Area Counts
C4H10	ppm	9.45	28912



Peak	Unit	Calibration Value	_Area Counts
CO2	ppm	10.00	7310

**Chromatogram 5% GeH4 in balance gas H2 measured with TCD**



Peak	Unit	Calibration Value	_Area Counts
GeH4	%	4.99	27067

Limit of detection (based on 3 times the noise level from a blank)

Components	Concentration (ppm)	Peak height	Noise	LDL (3x Noise)
Ar+O2	9.32	2312mV	0.28mV	3ppb
N2	9.69	2433mV	0.31mV	4ppb
CH4	10.0	2499mV	0.57mV	7ppb
CO	9.85	2435mV	0.48mV	6ppb
C2H6	10.0	2570mV	0.51mV	6ppb
C3H8	10.0	2571mV	0.47mV	5ppb
C4H10	9.45	2384mV	0.49mV	6ppb
CO2	10.0	2501mV	0.37mV	4ppb

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

**Repeatability:** iBased on the GC standards. Using 6 of 10 consecutive runs, being lower than 5% of 3\*CV%

**Linearity:** Based on the GC standards. A linear curve having its R2 at a value between 0.998 and 1.00.

**Accuracy:** Based on the GC standards. <= 1% of error or |dl whichever is higher

The MultiDetek3 detailed in this application note complies with the previously listed repeatability/linearity and accuracy standards.

## CONCLUSION

The MultiDetek3 configured with the PED and or TCD module offers a good analytical solution for trace ppb/ppm/% impurities for the validation of the quality and the production of germane gas product. The gas chromatograph is configured with standard industrial communication protocols and a remote-control interface. Due to its high sensitivity plasma emission detector, measuring trace impurities with the MultiDetek3 gas chromatograph down to sub ppb is perfectly suitable for the semiconductor grade germane. The MultiDetek3 is a very robust gas analyzer configured for industrial market to run 24/7. Combined with the other LDetek accessory modules, it fits the complete application requirements of the industry.

