High Purity Analyser

- Low level impurities in bulk gases
- Limit of detection: < 10 ppb
- ASTM D2504, D2505
- Also suitable for SiH_4, NH_3, SF_6, CF_4, HCl, Cl_2, GeH_4, N_2O
- Diaphragm valves with internal purge option for lower detection limits

*Get ready for **tomorrow's analytics***

Global Analyser Solutions

GAS offers custom configured GC analysers for many application fields for over 50 years. GAS analysers are designed to meet many standardised methods from GPA, ASTM, UOP, ISO, EN and others. The efficient configurations are based on proven GC technology, resulting in robust, highly productive instruments with an optimal return on investment.

Bulk quantity industrial gases are used in many application fields like pharma, petro, food, chemical and semi-conductors. Analysis of impurities is of prime importance for product quality. The GAS High Purity Analyser (HPA) is the standard tool for defining the exact specification of bulk gases.

Figure 1 High purity analyser (HPA) with external valve oven

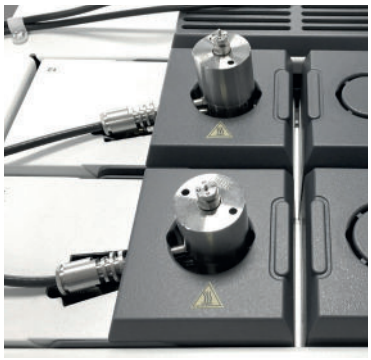

Figure 2 HPA on GC1600 with 2 Pulsed Discharge Detectors (PDD)

ppb detection limit in bulk gases

The GAS High Purity Analyser detects permanent gases like H_2, O_2, N_2, CH_4, CO, CO_2, noble gases and light hydrocarbons in a variety of bulk gases. The analyser reports low ppb concentration levels of the gases, which is required for purity specification. High sensitivity is achieved by utilising a Pulsed Discharged Detector (PDD); components of interest are measured after ionisation by helium discharge.

Diaphragm valves with extreme low leak rate

The high sensitivity imposes high demands to the analyser design and used parts. For this reason diaphragm valves are applied (figure 3), which offer an extreme low leakage rate and the best baseline stability compared to commonly used rotary valves. Figure 4 shows the principle of the internal purge option of the diaphragm valves: the purge grooves effectively isolate the process grove from ambient air, offering low leakage rate and avoiding cross port leaks.

Figure 3 Diaphragm valve with internal purge option provides millions of error-free injections

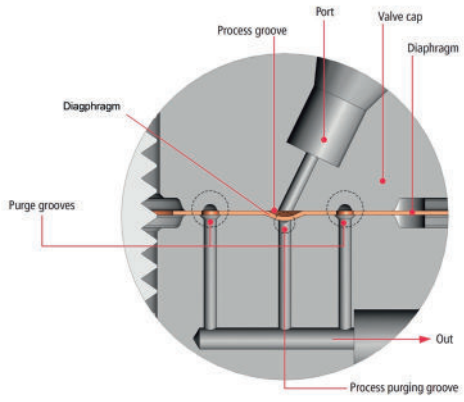

Figure 4 Internal purge option brings a highly leaktight system with a very low background signal

Results

Figure 5 and 6 show the chromatograms of trace levels of H_2, O_2, N_2, CH_4, CO and CO_2 using a 1/8" Molsieve column (5 ppm calibration standard in helium). Under these conditions, the limit of detection per component is:

H_2	< 20 ppb
O_2	< 10 ppb
N_2	< 10 ppb
CH_4	< 10 ppb
CO	< 20 ppb
CO_2	< 10 ppb

Figure 5 LOD's for chromatogram shown in figure 6

Managing the bulk gas

Managing the nearly 100% concentration level bulk gas is crucial in high purity analysis. The main component can disturb separation of the components or affect column performance. Therefore several options are available for venting or removing the bulk component:

- Backflush; e.g. analysing permanent gases in CO_2 or hydrocarbon streams
- Heartcut; e.g. analysis of N_2 in O_2
- Oxygen trap; for complete removal of O_2 bulk, e.g. measurement of Ar in O_2
- Argotek column, for separation of ppb Ar from O_2 bulk
- Plasma emission detector instead of PDD using Ar carrier gas in case of measurement of impurities in Ar bulk (figure 10).

Multiple channels

When the specified list of components require two or more separation columns, analysis channels can share a single PDD, or each channel is equipped with a separate PDD (figure 2) for parallel analysis.

Packed or capillary columns

HPA is available with packed or capillary columns, depending on required runtime and separation . Packed columns are sufficient for the separation of the components shown in figure 5 and 6.
A capillary column is used for Ar/O_2 separation, see figure 7.

Universal detector: many other components

PDD is a universal detector that can detect in principle every component except helium. Besides permanent gases also components like H_2S, COS, N_2O, NH_3 are successfully analysed at ppb levels (figure 9). HPA also measures impurities in bulk NH_3, SF_6, CF_4, HCl, Cl_2, SiH_4, GeH_4, N_2O, Xe, Kr.

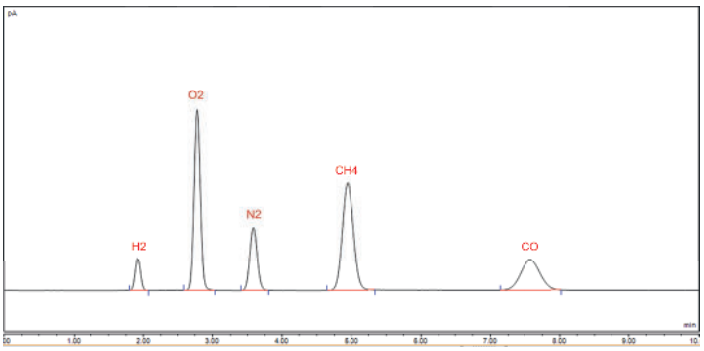

Figure 6 Chromatogram HPA. 5 ppm standard in He

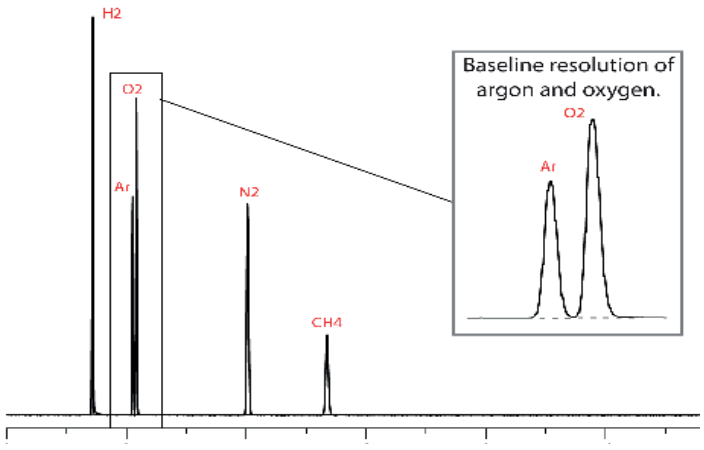

Figure 7 Chromatogram HPA. A capillary column offers Ar/O2 separation

Inj. No.	Injection Name / Selected Peak:	Area pA*min				
		BackDetector Hydrogen	Oxygen/Argon	Nitrogen	Methane	Carbon monoxide
76	HP mix repeatability	19.1419	128.0336	52.5880	137.3778	66.9799
77	HP mix repeatability	19.0546	127.7394	52.2930	137.8404	66.8278
78	HP mix repeatability	19.1606	127.8858	52.2376	138.1658	66.8532
79	HP mix repeatability	19.2118	128.1375	52.3762	138.6659	67.4800
80	HP mix repeatability	19.2232	128.5649	52.1210	138.9492	67.4995
81	HP mix repeatability	19.2887	128.2764	52.2102	138.5774	67.0324
82	HP mix repeatability	19.2745	128.1268	52.3552	139.0833	67.7506
83	HP mix repeatability	19.3573	128.4626	52.1678	138.3944	67.1328
84	HP mix repeatability	19.3072	128.4414	52.3882	138.6392	66.7665
85	*HP mix repeatability*	*19.2289*	*128.3447*	*52.2726*	*138.4434*	*67.1574*
Maximum		19.3573	128.5649	52.5880	139.0833	67.7506
Average		**19.2249**	**128.2013**	**52.3010**	**138.4137**	**67.1480**
Minimum		19.0546	127.7394	52.1210	137.3778	66.7665
Standard Deviation		0.0889	0.2656	0.1341	0.5100	0.3289
Relative Standard Deviation		**0.46%**	**0.21%**	**0.26%**	**0.37%**	**0.49%**

Figure 8 Repeatability. 5 ppm standard in Helium

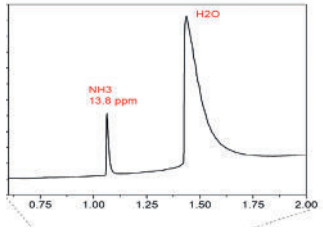

Figure 9

Low ppm ammonia in hydrogen. PDD detector. LOD is 100 ppb

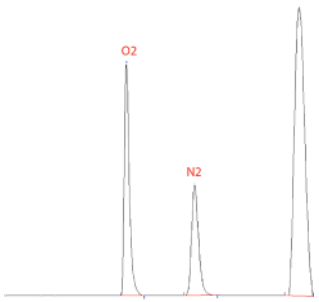

Figure 10

Oxygen analysis in argon using Plasma emission detector. Since argon is used as carriergas, bulk argon is not visible

Specification

Standardised methods:	ASTM D2504, D2505
Configuration:	1-4 channel instrument based on Thermo Trace GC1600 or CompactGC$^{4.0}$
	Detector: Pulsed Discharge Detector (PDD)
	Injection: GSV
	Columns: packed, wide-bore or capillary
Optional:	- Backflush option
	- Heartcut option
	- DEOX facility for complete trapping of O_2 bulk
	- Argotek column for complete separation of ppb Ar in bulk O_2
	- Vaporiser or Pressure Facility for LPG samples
	- Plasma Emission Detector for impurities in argon
Sample tubing:	Sulfinert® or Hastelloy C tubing for inert sample path
Application:	Custom configured analyser for the analysis of permanent gases (ASTM D2504, D2505) and other components in various bulk gases like permanent gases, hydrocarbons streams, SiH_4, NH_3, SF_6, CF_4, HCl, Cl_2, GeH_4, N_2O Xe, Kr
Range:	low ppb - 200 ppm, depending on the component
Detection limit:	see figure 5
Repeatability:	3 % RSD or better, depending on concentration level and separation
Sample requirements:	See our pre-installation guide for additional requirements

Figure 11 HPA on CompactGC$^{4.0}$