AFP
ANALYTICAL FLOW PRODUCTS

AN-02
Application Note-02

MEDIUM PRESSURE GAS CHROMATOGRAPHY (MPGC)

Rev. 2.0

It is a well know fact that the adsorption in a GC column packing material, behaves differently based on local pressure conditions. This is true for gas solid chromatography (GSC) system, where the separation mechanism is mainly governed by surface adsorption. However, great care is taken to avoid this effect in gas-liquid chromatography (GLC). This can be seen in various manufacturer's adsorption isotherm curve. To simplify this phenomenon, applying a vacuum releases the ''Trapped'' molecules from it structure; inversely pressurized, it increases adsorption. Furthermore, in presence of many impurities, the relative adsorption will change based on operating pressure.

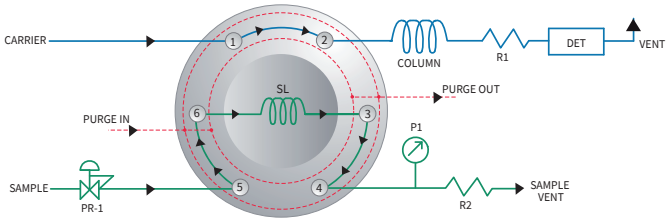
The common 1/8'' O.D. pack columns are still widely in use today, in gas solid chromatography. The reason for this, is the large sample volume that can be injected resulting in a much greater sensitivity, than achievable with capillary and plot column. However, 1/8''pack columns have a poor resolution and extremely low HETP. The proposed method will improve the separation performance for such columns. It is also possible to improve sensitivity as it will be demonstrated below.

Most of the time in gas chrmatography, the outlet of the column is at or near the atmospheric pressure, depending on particular system chromatographic configuration. This means that carrier gas velocity increases when approaching the column's end. Accordingly, the carrier gas pressure is decreasing and also adsorption.

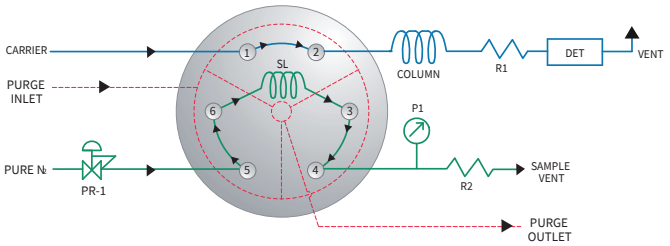
Operating the column outlet at a higher pressure will increase the relative adsorption of various components, resulting in enhanced selectivity or peak separation.

This is what MPGC is all about, i.e. operating the system at higher pressure. This is simply realized by adding a restriction between the column outlet and the detector, in order to increase outlet column pressure. Obviously, this also increases the inlet pressure.

MDVG diaphragm series



Rotary CLP series



At the same time, if the sample pressure is high enough, a restriction could also be added on sample vent, in order to increase the pressure in the sample loop. Doing so, allows a reduction of the length of the sample loop while keeping the impurities level high enough for the target system detection limit. This helps to eliminate a difficult to-separate background, improve heartcut, or backflush and increase lifetime of various catalyst of palladium base H2 membrane separation that could be used in such system.

Many chromatographers have tried to increase sample pressure to improve the detection limit (LDL) of their detector (Ex. for a TCD). However, due to existing chromatographic valve leak rates (diaphragm or rotary), the result they got was the opposite of what they expected.

This is due to the fact that when sample pressure was increased, the sample gas was leaked into carrier gas, killing detector sensitivity and/or separation. So, the only solution was to keep carrier pressure higher than the sample pressure. This resulted in another drawback, since carrier begin to leak into sample, resulting in a detrimental effect on repeatability.

One way or the other, most of GCs are configured this way. The system performance is dramatically limited by the valve. In order to use our proposed method, GC valves must have a high sealing integrity. The AFP™ valve family allows for such an operation for an extended period of time without system performance degradation. **Increasing the column and sample pressure will improve the overall system performance, at the expense of only two flow restrictors.**

Which pressure will be the best for my system?

This is a good question and it is something that one may calculate based on manufacturer thermodynamics adsorption data. There is a good text book information on this. However, the use of a simple needle valve acting as variable orifice during system configuration will save a lot of precious time. When proper needle valve setting has been found, it may easily replaced by a flow restrictor made of a small piece of 1/16'' OD, SS tubing.

The GC valve may be from the MDVG or DV family. The CLP type of the RV rotary valve family could also be used with excellent results. All these valves family have pressure required level of sealing integrity.

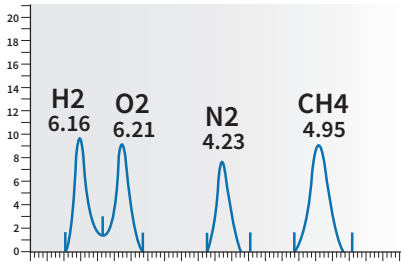
The following chromatograms show examples of improved separation and sensitivity that can be achieved with the use of AFP™ valves and this method. Our technique of MPGC can add significant improvements to your gas solid chromatography. Generally speaking, the use of high performance valves will always improve the performance of any system.

Benefits :

- **ENHANCES PEAK SELECTIVITY**
- **IMPROVES BACKGROUND SEPARATION**
- **IMPROVES DETECTOR SENSITIVITY**
- **ALLOWS SHORTER COLUMN LENGTH AND HIGHER TEMPERATURE OPERATION**

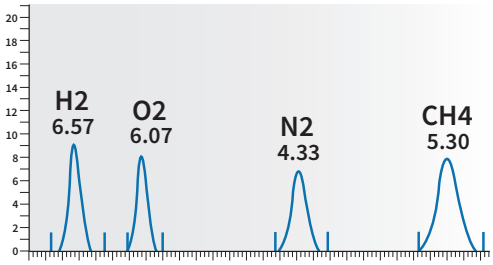
Column outlet and sample pressure at atmospheric pressure. Traditional method

1 -

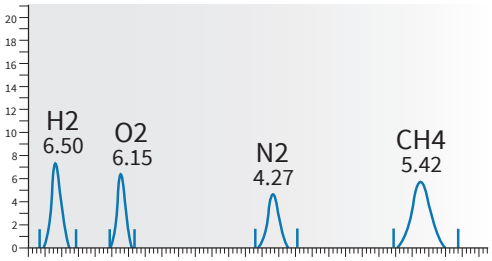


Column outlet pressurized

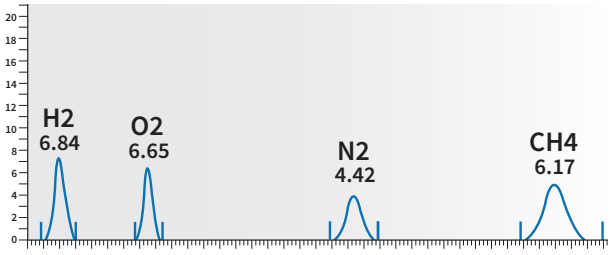
2 - COLUMN OUTLET PRESSURIZED AT 10 PSI (69 KPA)



3 - COLUMN OUTLET PRESSURIZED AT 20 PSI (138 KPA)

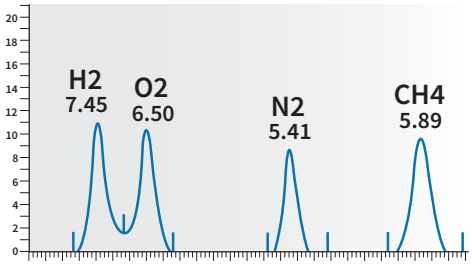


4 - COLUMN OUTLET PRESSURIZED AT 40 PSI (276 KPA)

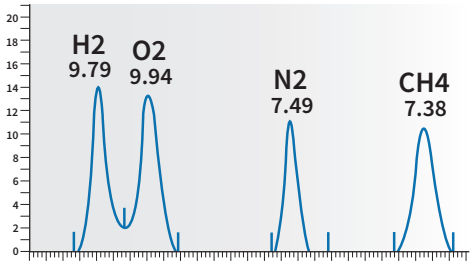


Pressurized sample

5 - SAMPLE LOOP AT 10 PSI (69 KPA), COLUMN OUTLET PRESSURE AT ATMOSPHERIC

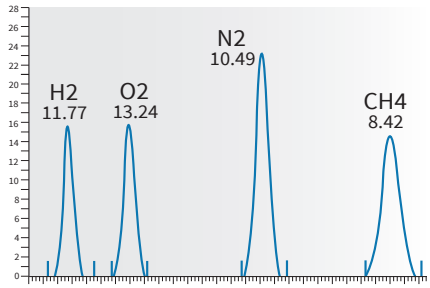


6 - SAMPLE LOOP AT 20 PSI (138 KPA), COLUMN OUTLET PRESSURE AT ATMOSPHERIC

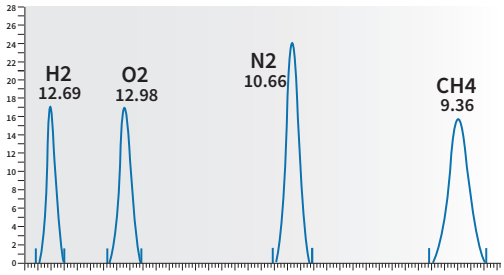


Combined effect

7 - SAMPLE LOOP AT 30 PSI (207 KPA), COLUMN OUTLET AT 10 PSI (69 KPA)

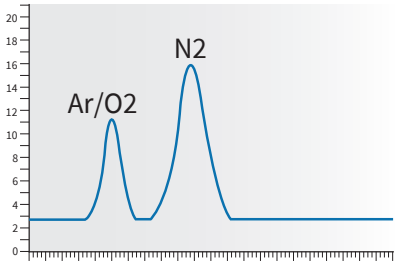


8 - SAMPLE LOOP AT 40 PSI (276 KPA), COLUMN OUTLET AT 40 PSI (276 KPA)

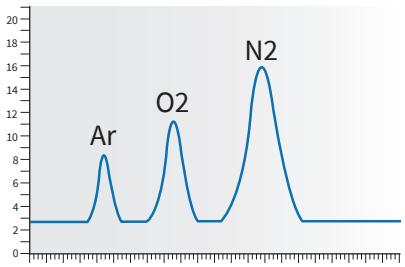


Other application examples

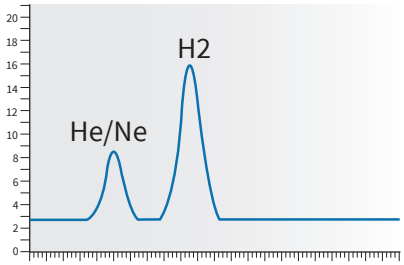
1 - STANDARD CONFIGURATION CHROMATOGRAM OF AIR ON A HEAT TREATED MS 5A COLUMN, TEMP. 25°C. HELIUM AS CARRIER.



2 - SAME OPERATING CONDITION WITH MPGC CONFIGURATION



3 - STANDARD CONFIGURATION HE/NE/H2 MIXTURE ON A HEAT TREATED MS 5A COLUMN, TEMP. 25°C. ARGON AS CARRIER.



4 - SAME OPERATING CONDITION WITH MPGC CONFIGURATION