

Fast Trace Analysis in Ultra High Purity Helium using Parallel PLOT Columns and a Single PDHID Detector



Application Note

AN0007

INTRODUCTION

Low level detection of permanent gases and carbon dioxide is essential for Ultra High Purity helium since traces of these components affect the quality and thus the value of the helium gas. The quantification of trace gases is highly applicable using Gas Chromatography (GC) due to the complete separation of the analytes prior to detection, and in addition, the non-sensitivity of the Pulsed Discharge Helium Ionization Detector (PDHID) to helium.

Performing a complete separation of permanent gases and carbon dioxide by GC is not possible using a single column. The common solution is a configuration of two identical analytical channels with the two columns having a different type of stationary phase. By optimisation of the column dimensions, it is possible to combine the two columns into one channel. This application note describes the trace analysis of permanent gases and carbon dioxide in helium using a single channel configuration with two columns connected in parallel.

EXPERIMENTAL

The SCION 456-GC configuration consisted of two parallel columns for the optimisation of a fast separation of permanent gases. The sample is injected by a gas sampling valve via a mini-gas splitter to two columns, as illustrated in Figure 1.

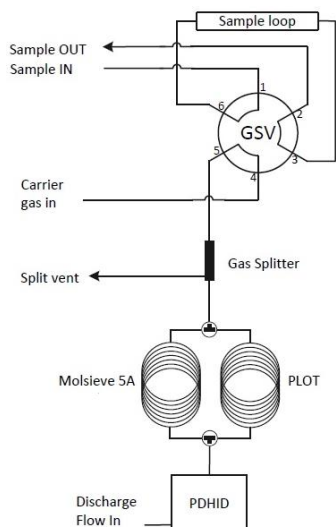


Fig 1. Schematic Illustration of Parallel Column GC System

A SCION-Molsieve 5A column is used to separate hydrogen, oxygen, nitrogen, methane and carbon monoxide before the composite air peak elutes from the SCION PLOT-Q column. After this composite peak, the methane and carbon dioxide elute from the SCION PLOT-Q column. Column choice was optimised to ensure that all permanent gases elute before the t_0 time of column 1, preventing any mixed elution from both columns.

Table 1 shows the analytical conditions of the SCION GC.

Table 1. Analytical Conditions

Conditions	
Injector	Gas Sampling Valve, Mini Gas Splitter 1:15
Column 1	SCION Molsieve 5A 10m x 0.32mm x 30 μ m
Column 2	SCION PLOT Q 30m x 0.32mm x 10 μ m
Oven Programme	60°C Isothermal
Carrier Gas	Helium, 99.99999%, 12psi
FID	120°C, Range 12

RESULTS

When using PDHID as a detection technique with the parallel column setup, the permanent gases and carbon dioxide can be analysed down to a lowest detection limit (LDL) of approximately 50ppb within 120 seconds. With the high inertness of the SCION molsieve column, all components including carbon monoxide elute with a perfect peak shape allowing low detection levels, as shown in Figure 2.

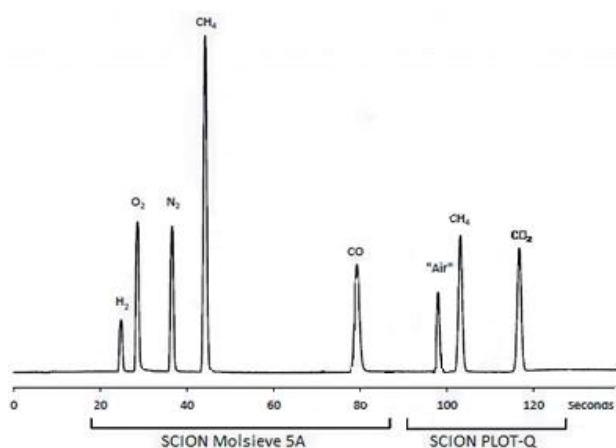


Fig 2. All components at a concentration of 2ppm in Helium

As methane elutes from both columns it was used as a component to validate the analytical performance, between the two columns. The repeatability results are all less than 1.0% RSD, over 20 consecutive GC runs. To validate the concept of parallel column analysis, the repeatability of methane from both columns is used. The repeatability of methane from the individual columns (Molsieve RSD 0.65%, PLOT RSD 0.72%) is very similar to the repeatability of the summed methane (RSD 0.66%). Figure 3 shows the percentage of methane injected on the two columns. The amount of methane entering the Molsieve column is 67% with 33% entering the PLOT column.

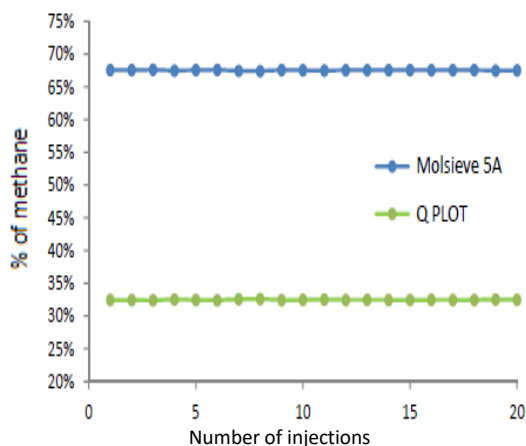


Fig 3. The repeatability of methane (n=20)

Table 2 shows the mean, standard deviation and relative standard deviation (RSD%) of low level trace gases in the helium, at the 2ppm level with 20 injections. The excellent repeatability proves the working concept of parallel columns where the sample is split between two columns.

Table 2. Repeatability data of parallel column system

Column	Analyte	Mean (ppm)	Std Dev	RSD%
Molsieve	H ₂	2.0	0.015	0.73
	O ₂	2.0	0.012	0.59
	N ₂	2.0	0.011	0.57
	CH ₄	2.0	0.013	0.65
	CO	2.0	0.007	0.34
Q PLOT	"Air"	2.0	0.007	0.67
	CH ₄	2.0	0.014	0.72
	CO ₂	2.0	0.010	0.48
	Summed CH ₄ Area	340787	2266	0.66

Conclusions

The SCION 456 GC configured with parallel column setup with the use of the PDHID detector ensures trace levels of permanent gases and carbon dioxide are analysed within 120 seconds, down to an LDL level of 50ppb. The validity of the instrumentation was shown through exceptional repeatability when parallel columns with a single injector and detector system is used for high purity helium analysis.