「Application Note」

Analysis of Natural Gas by ChroZen GC According to ASTM D1945

- GC Application

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Abstract

Natural gases consist of methane (major component), other hydrocarbons and permanent gases like hydrogen, oxygen, nitrogen as well as carbon dioxide. They are used as major energy sources in numerous industries and it is very critical to accurately determine them because their value differs depending on chemical composition and the concentration of each component.

Among various analytical methods for natural gases, ASTM D1945 utilizes Gas Chromatograph (GC) configuring 5 columns and 3 detectors with 4 valves, which enables the analysis of complex natural gases in a single injection. (Fig. 1)

ChroZen GC with column switching valve system effectively analyzes natural gases by controlling the valve switching time with accuracy and precision and the gas flow can easily be modified depending on the gas composition.

In this study, ChroZen GC verifies its reliability by effectively determining each component of natural gases by one GC according to ASTM D1945.

Instruments and Software

item	Description	Part No.	Unit
Oven	ChroZen GC Mainframe Assembly with UPC Detector Board Unit	6701012502	1
Inlet	Capillary Inlet Assembly for ChroZen GC	6701012550	1
	Packed Inlet Assembly for ChroZen GC	6701012510	2
Detector	FID Assembly for ChroZen GC	6701012590	1
	TCD (Thermal Conductivity Detector) Assembly for ChroZen GC	6701012570	2
CDS	YL-Clarity software for single instrument of YCM GC	5301011020	1
Column	GS-gaspro 60 m 0.32 mm	-	1
	2m 1/8 2mm MOLECULAR SIEVE 5A 60/80	-	2
	6Ft Porapak Q 60/80 SS	-	2
Install. Option	Start-up kit	1601011110	1
Valve	Automatic Gas Valve, 2 pos/6 port, Valcon E/Max: 225°C for ChroZen GC Micro-electric actuator type with 250 ul sample loop	6501011250	2
	Automatic Gas Valve, 2 pos/10 port, Valcon E/Max: 225°C for ChroZen GC Micro-electric actuator type with 250 ul sample loop	6501011280	2

Fig 1. ChroZen GC with Column Switching Valve System

Analytical Condition

GC condition	
Oven	50°C (4 min) -> 30°C / min -> 90°C (3 min) -> 15°C / min -> 150°C(8.0 min)
Inlet 1 - Capillary	200°C
	GS-gaspro 60 m 0.32 mm / Flow : 5 mL/min (Helium) / split 1:10
Inlet 2 - Packed	150°C
	2m 1/8 2mm MOLECULAR SIEVE 5A 60/80 Flow : 20 mL/min 6Ft Porapak Q 60/80 SS (Helium)
Inlet 3 - Packed	150°C
	2m 1/8 2mm MOLECULAR SIEVE 5A 60/80 Flow : 20 mL/min 6Ft Porapak Q 60/80 SS (Argon)
Detector 1 – FID	250°C Air – 300 mL/min Makeup – 20 mL/min Hydrogen – 30 mL/min
Detector 2 – TCD 1	150°C Reference – 30 mL/min
Detector 3 – TCD 2	150°C Reference – 30 mL/min Makeup – 20 mL/min

Valve program				
Time(min)	Valve1 (*EV / 10 port)	Valve2 (EV / 10 port)	Valve3 (EV / 6 port)	Valve4 (EV / 6 port)
0.1	On	On	Off	Off
0.9	Off	-	-	-
1.8	-	-	-	On
6.5	-	-	On	-
10.1	-	-	-	Off
Valve temperature	100°C			

* μ-Electric Actuator Valve

Summary of Test Method

As instructed by the standard method for Natural Gas Analysis (NGA), ASTM D1945, the analysis is conducted by ChroZen GC with 3 detectors (1 FID and 2 TCDs), 5 columns (Molesieve 5A/Porapak Q/Gaspro) and switching valve system. (Fig 2)

Valve 1

The Valve 1 is connected to Molsieve 5A and Porapak Q column each. The loaded gas sample is transfered to the Porapak Q column by carrier gas(Ar) and initially eluted light compounds such as He, H_2, O_2, N_2 go to the Molsieve 5A column to be separated. The rest of gas sample is vented by switching the column flow.

Valve 2

There are 2 sample loops installed in Valve 2 and the gas sample loaded in sample loop 1 goes to Gaspro column. Hydrocarbons including CH_4 are separated in it and these are detected by FID. The gas sample loaded in sample loop 2 goes to Valve 3 installed with Porapak Q column and Valve 4 installed with Molseive 5A column to be detected by TCD.

Valve 3

The Valve 3 is for backflush to detect Ethane (C_2H_6) and H_2S with higher effect. It requires high oven temperature to elute C_2H_6 and H_2S from Porapak Q column but this temperature might cause co-elution of O_2, N_2, CH_4 and CO in Molsieve 5A column connected to Valve 4. So, C_2H_6 and H_2S need to be back-flushed at the relatively low temperature.

Valve 4

The Valve 4 is to trap O_2, N_2, CH_4 and CO in Molsieve 5A column while the remained hydrocarbons are back-flushed from the Porapak Q column. Molsieve 5A column separates H_2, O_2, N_2, CH_4, CO in order but the compounds heavier than CO can easily be adsorbed in the column and not eluted, it's important for those compounds not to flow into the column. It will be too late to switch the flow after eluting CO from Molsieve 5A column because those compounds would already be adsorbed in the column. Therefore, it's better to switch the flow (Valve 4 Off) right after H_2 elution and capture O_2, N_2, CH_4 and CO until C_2H_6 and H_2S are back-flushed from Porapak Q. Then, the valve 4 is turned to on- position to elute O_2, N_2, CH_4 and CO from Molsieve 5A column to be detected by TCD.

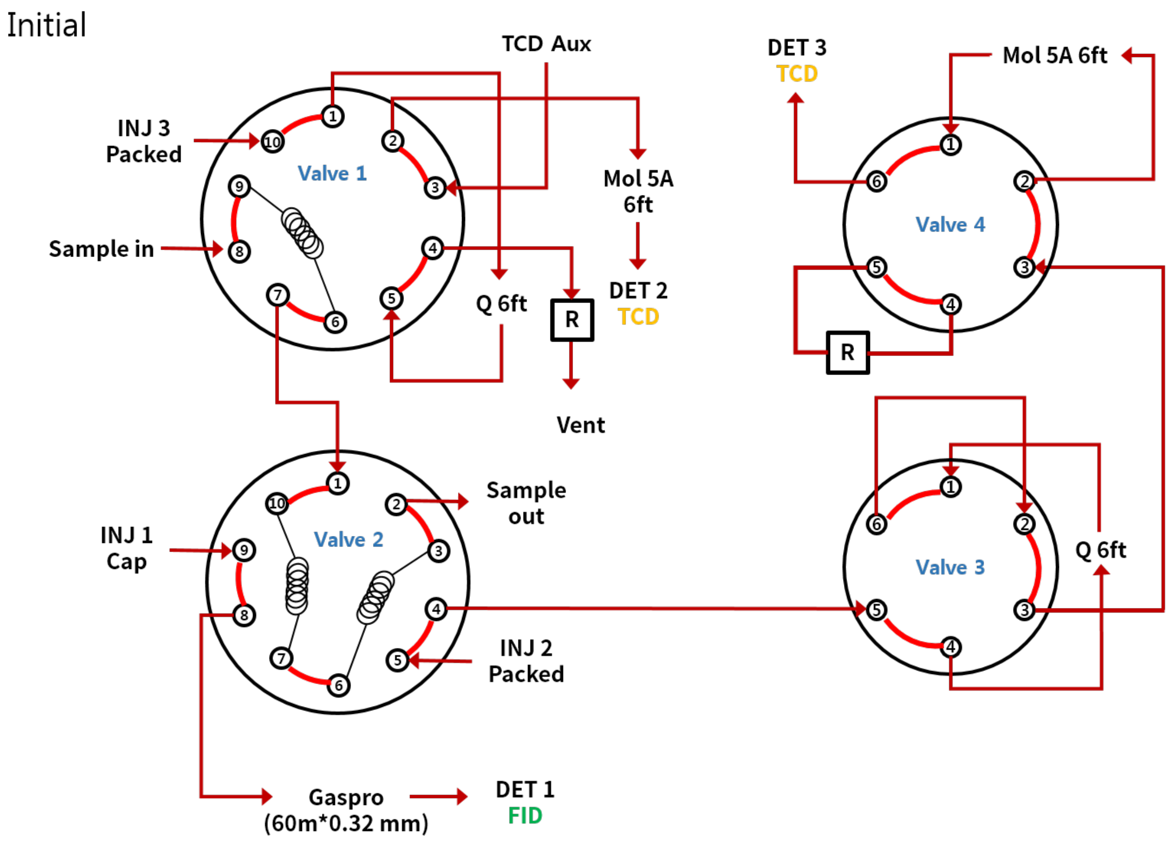
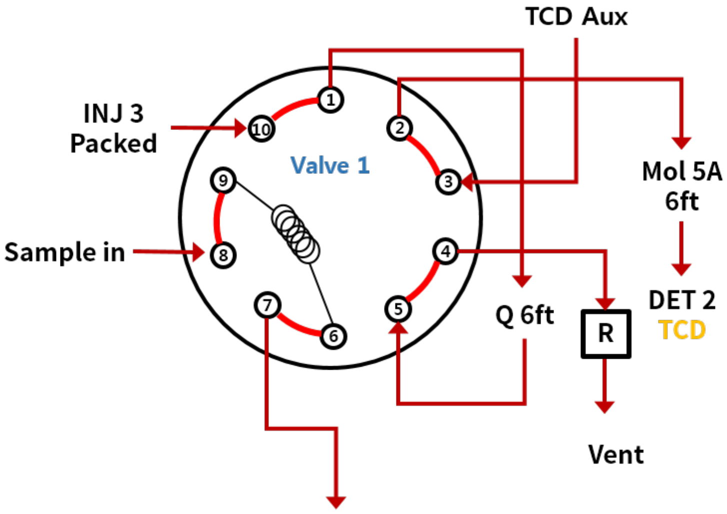
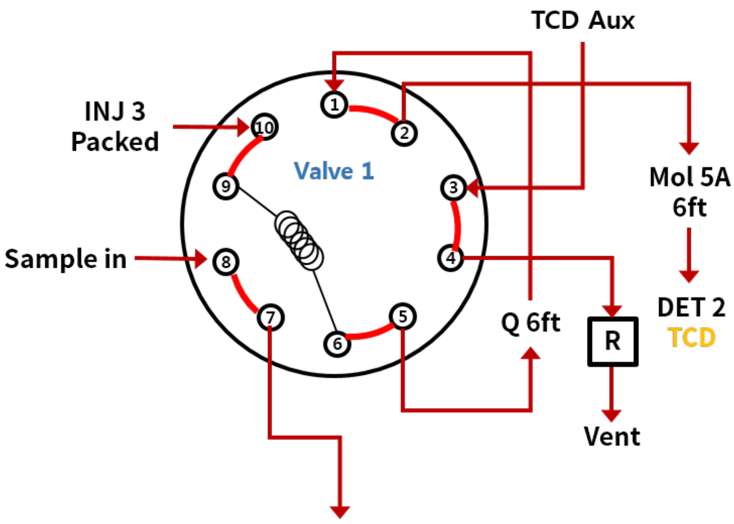


Fig 2. Valve Diagram of Natural Gas Analysis

Valve 1 Switching Time Setting

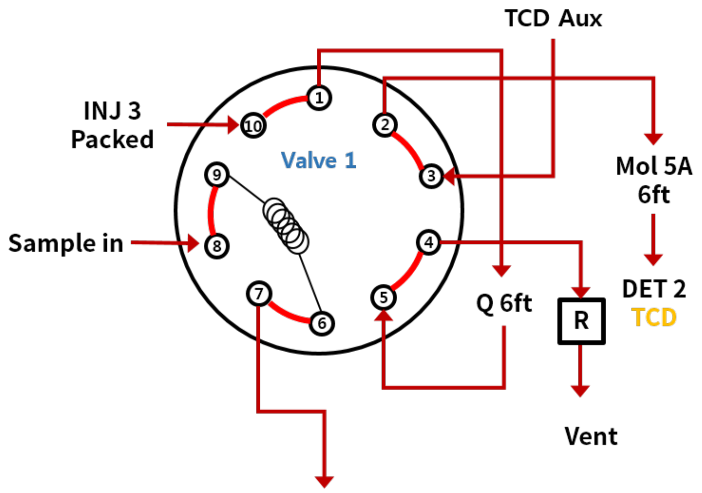


The sample is loaded in the sample loop installed on the Valve 1. (Valve 1-Off)



Turn the Valve 1 to on-positon at 0.1 min after the analysis run to transfer the sample loaded in the sample loop to Porapak Q column and then Molsieve 5A column to be detected by TCD. (Valve 1-On)

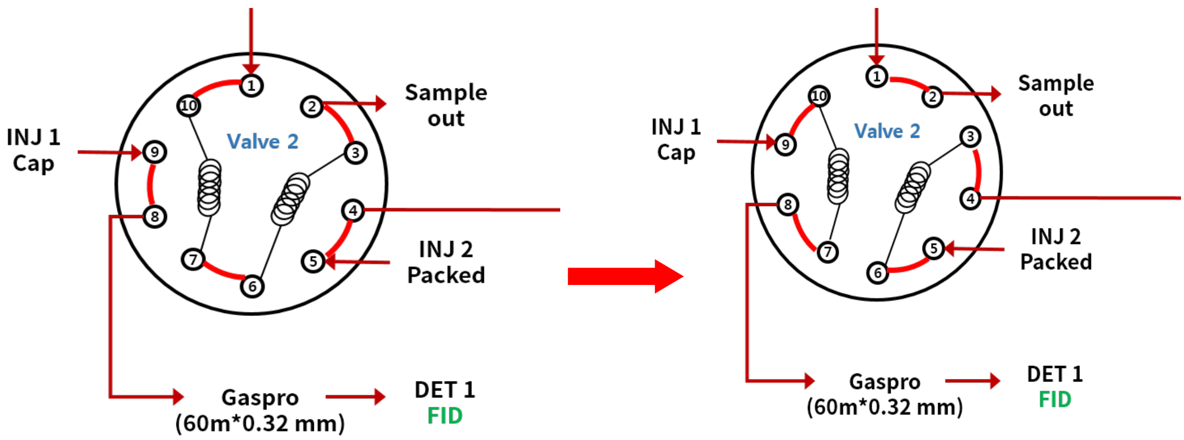
Time(min)	Valve1 (EV / 10 port / 250 µL)	Valve2 (EV / 10 port / 250 µL)	Valve3 (EV / 6 port)	Valve4 (EV / 6 port)
0.1	On	On	Off	Off



After H2 compound completely eluted from the molsieve column, turn the valve 1 to off - positon to vent the hydrocarbon groups remained in Porapak Q column not to be transferred to Molsieve 5A column. (Valve 1- Off)

Time(min)	Valve1 (EV / 10 port / 250 μL)	Valve2 (EV / 10 port / 250 μL)	Valve3 (EV / 6 port)	Valve4 (EV / 6 port)
0.1	On	On	Off	Off
0.9	Off	-	-	-

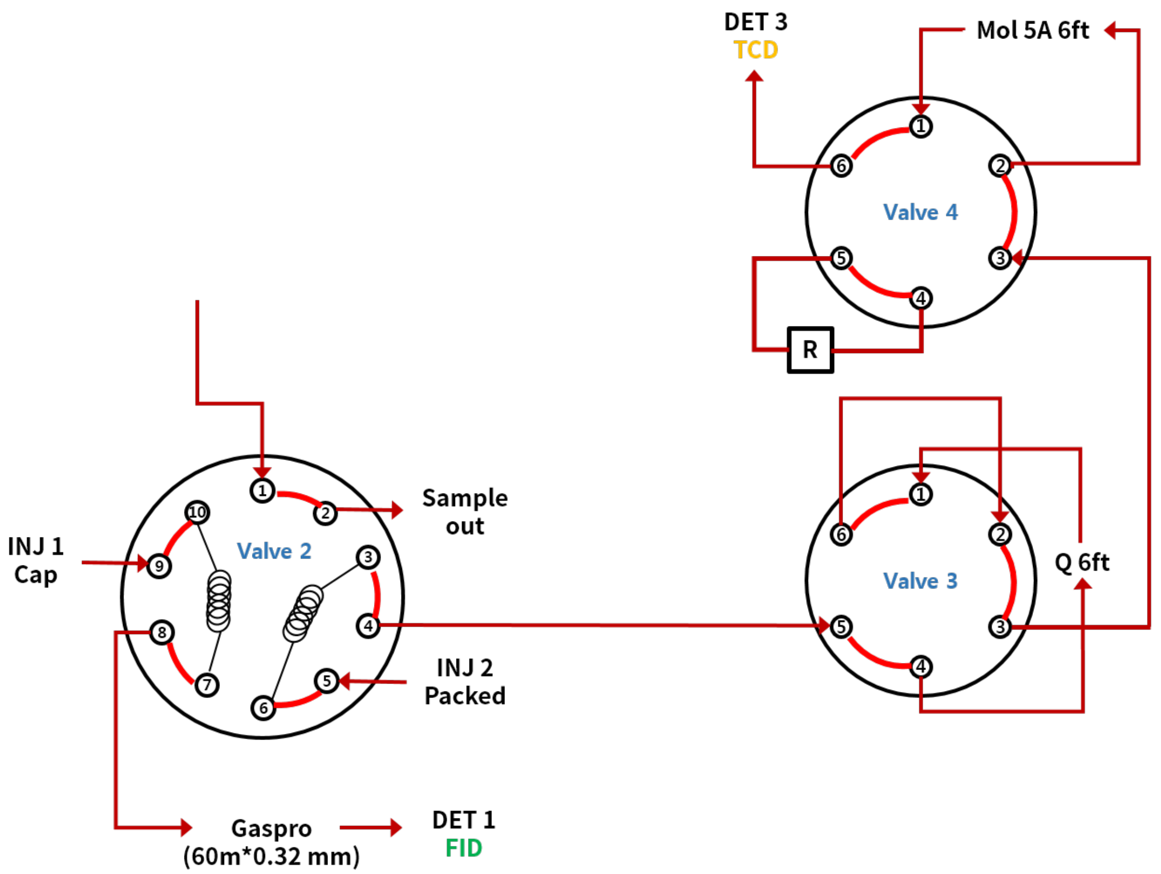
Valve 2 Switching Time Setting



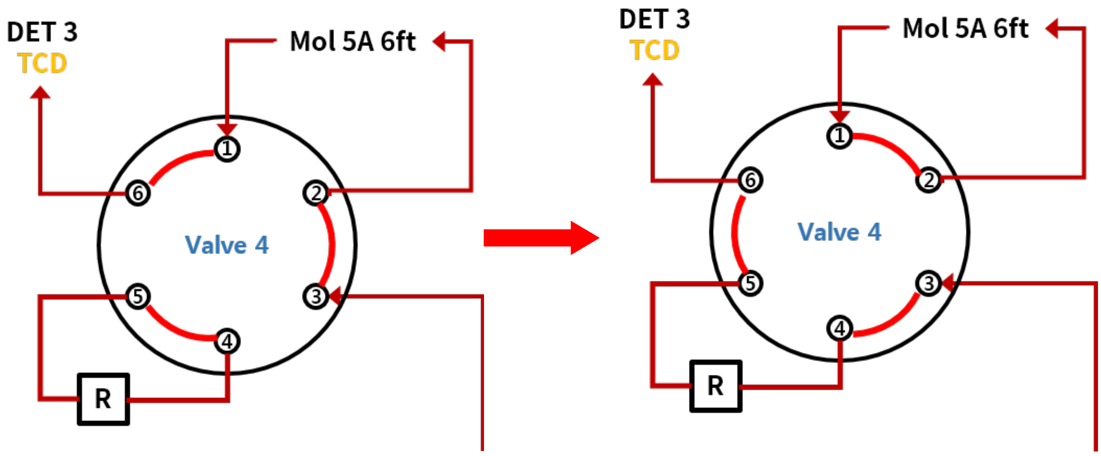
There are 2 sample loops installed in Valve 2 and same samples are loaded in each sample loop. Set the valve switching time to On position at 0.1min. (Valve 2-On)

Time(min)	Valve1 (EV / 10 port / 250 µL)	Valve2 (EV / 10 port / 250 µL)	Valve3 (EV / 6 port)	Valve4 (EV / 6 port)
0.1	On	On	Off	Off

Valve 3, 4 Switching Time Setting



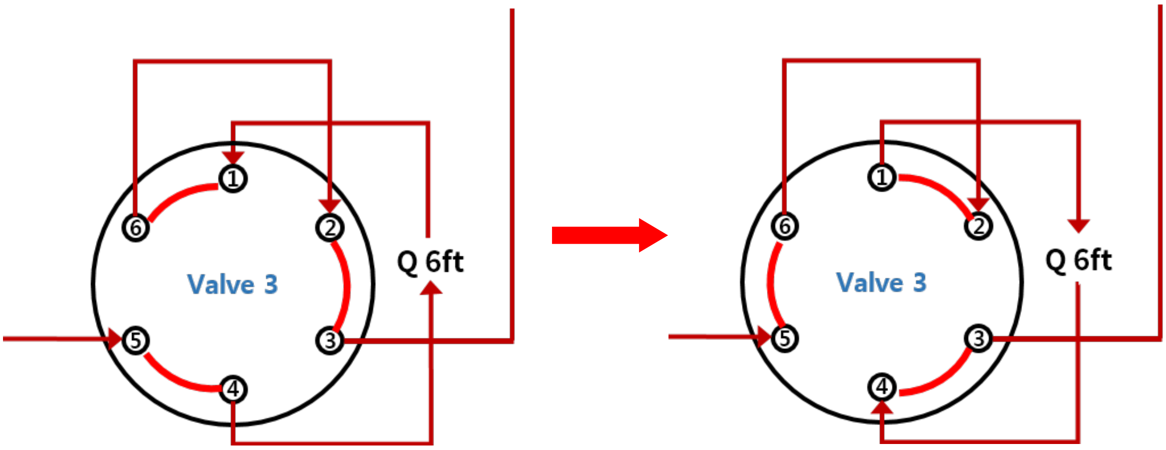
When the sample loaded in the sample loop on Valve 2 is injected 0.1 min after analysis run, it is transferred to Porapak Q column connected with Valve 3. (Valve 3-Off)
The compounds eluting after CO_2 are remained in Porapak Q column while others such as He, H_2, O_2, N_2, CH_4 and CO are co-eluted in advance.



He, H_2, O_2, N2, CH_4 and CO are transferred to Molsieve 5A column with Valve 4 and separated in it. It's important to switch the flow right after H_2 elution and trap O_2, N_2, CH_4 and CO in Molsieve 5A column. (Valve 4-On)

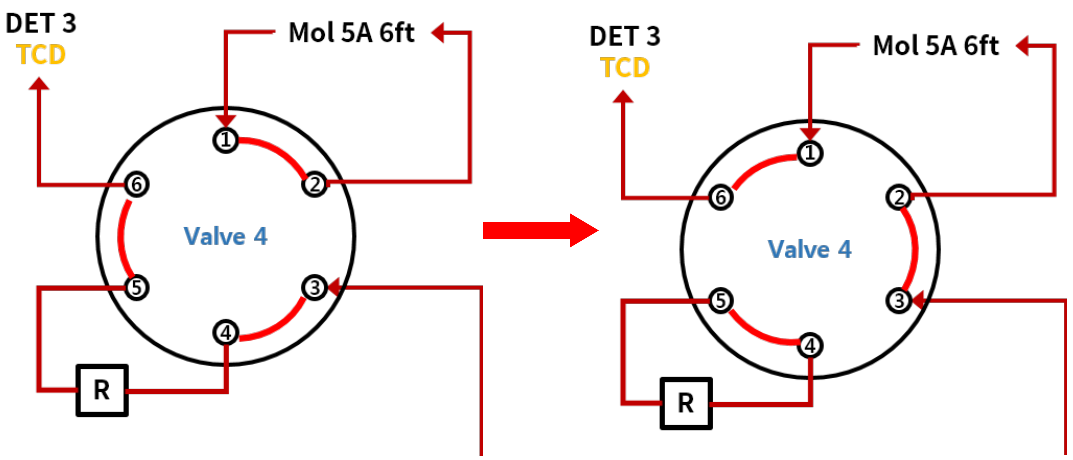
If not, it will be too late to switch the flow after eluting CO from Molsieve 5A column because those heavy compounds would already be adsorbed in the column.

Time(min)	Valve1 (EV / 10 port / 250 µL)	Valve2 (EV / 10 port / 250 µL)	Valve3 (EV / 6port)	Valve4 (EV / 6port)
0.1	On	On	Off	Off
0.9	Off	-	-	-
1.8	-	-	-	On



It requires high oven temperature to elute heavy hydrocarbons like C_2H_6 from Porapak Q column but this high temperature might cause co-elution of O_2, N_2, CH_4 and CO in Molsieve 5A column connected to Valve 4. So, C_2H_6 and H_2S need to be back-flushed at the relatively low temperature. (Valve 3-On)

Time(min)	Valve1 (EV / 10 port / 250 µL)	Valve2 (EV / 10 port / 250 µL)	Valve3 (EV / 6 port)	Valve4 (EV / 6 port)
0.1	On	On	Off	Off
0.9	Off	-	-	-
1.8	-	-	-	On
6.5	-	-	On	-



After that, turn the Valve 4 off-position to separate O_2, N_2, CH_4 and CO in Molsieve 5A column to be detected by TCD.

Time(min)	Valve1 (EV / 10 port / 250 µL)	Valve2 (EV / 10 port / 250 µL)	Valve3 (EV / 6 port)	Valve4 (EV / 6 port)
0.1	On	On	Off	Off
0.9	Off	-	-	-
1.8	-	-	-	On
6.5	-	-	On	-
10.1	-	-	-	Off

Gas mixture analysis

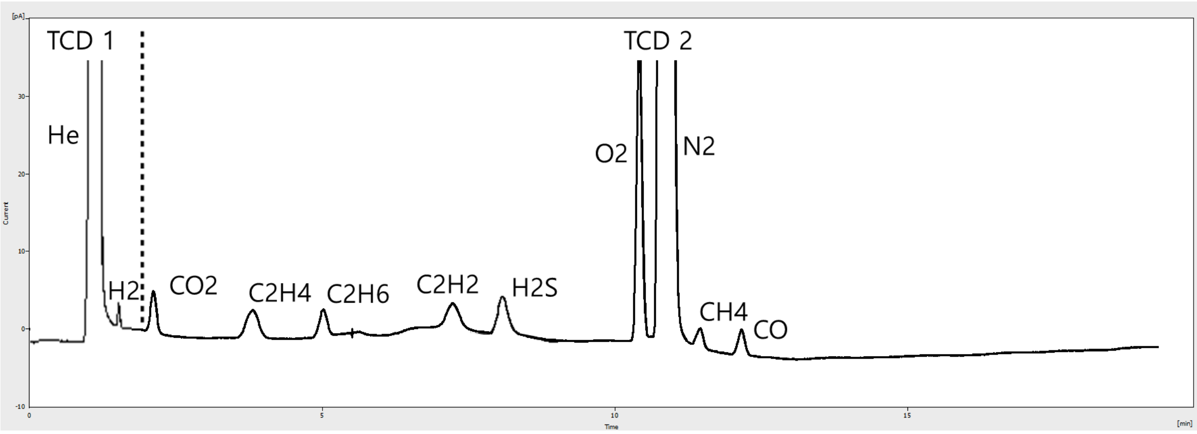
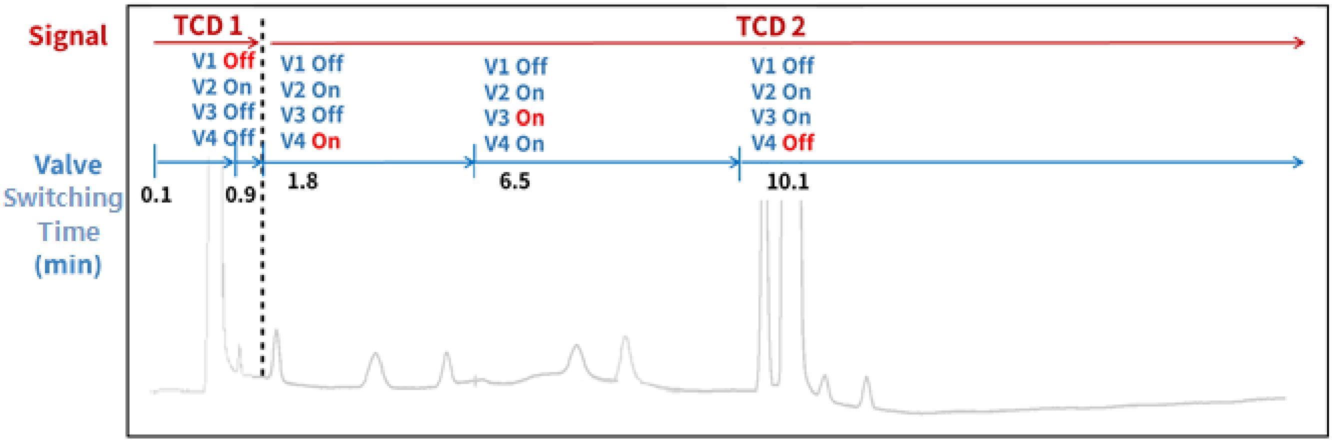
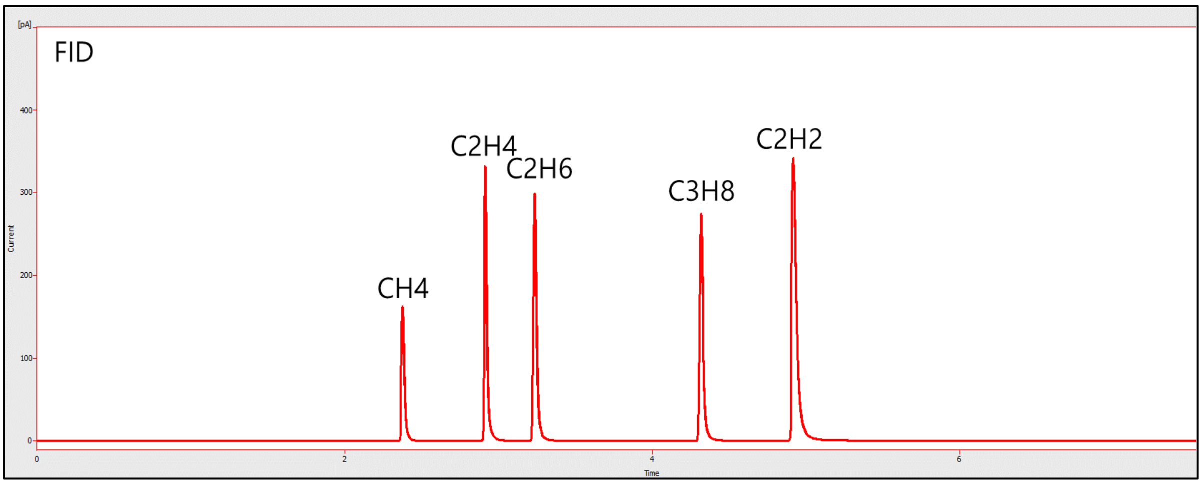


Fig 3. Chromatogram of Natural Gas Mixture





These results show complete separation and detection of each compound in natural gas mixture by ChroZen GC configuring 5 columns and 3 detectors with 4 valves.

Conclusion

In this study, the determination of the chemical composition of natural gas mixture was conducted by ChroZen GC configuring 5 columns and 3 detectors with 4 valves according to ASTM D1945.

Also, it’s easy to modify the valve configuration depending on the target gaseous sample and figure out the optimized valve switching time.

As the results, ChroZen GC verifies the right solution for superior resolution and sensitivity of components in natural gases within the range of composition shown in ASTM D1945.

Reference

- ASTM D1945 Standard Test Method for Analysis of Natural Gas by Gas Chromatography

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