

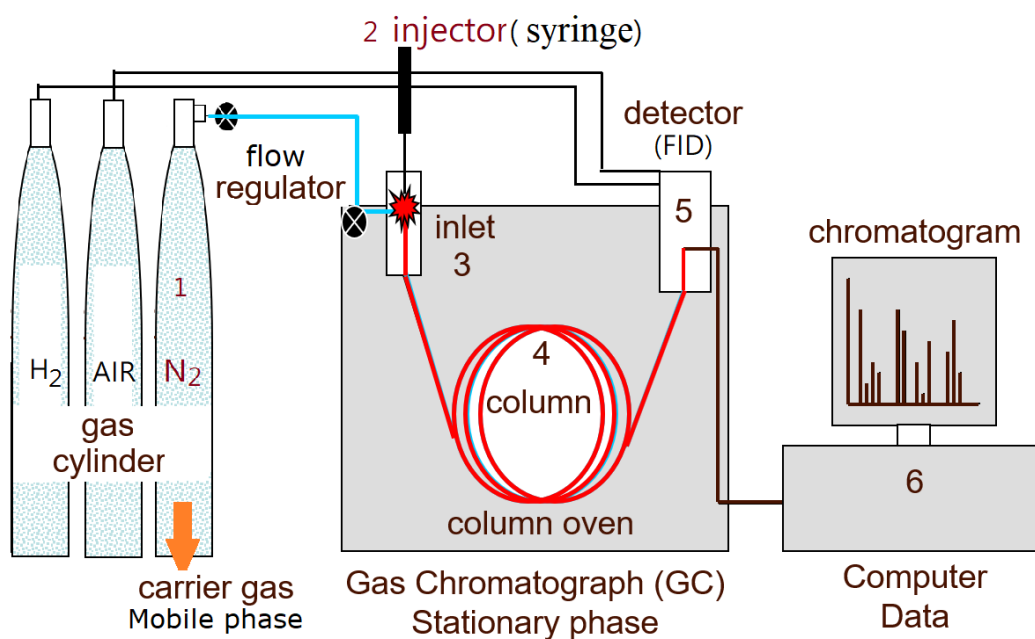
Experiment 10 Gas Chromatography of Alcohols

Gas chromatography (GC) is a technique used to analyze mixtures. The instrument allows mixtures to be separated and the amount of each component to be determined. One advantage of this technique is that very small (a few microliters) samples can be analyzed.

Samples to be analyzed in a gas chromatograph must be volatile, that is, they must vaporize easily. Once vaporized, the sample is carried through a column containing a porous material. A nonreactive gas, such as He or N₂ is used to carry the components of the mixture through the column. Not all components of a mixture travel through the column at the same rate. Thus, some components will arrive at the detector at the end of the column before others. As the components pass over the detector, the detector sends a signal to a recorder and a chromatogram is produced.

Using the chromatogram, the percent composition (amount) of each component in the mixture can be determined. The percent composition is directly related to the area of each peak in the chromatogram.

In this investigation you will use gas chromatography to separate the contents of an alcohol mixture and then determine the components in the mixture of the alcohols.



GC Schematic Diagram

Instrument

GC: Shimadzu GC-2014

Carrier gas : N₂

Detector : Flame Ionization Detector (FID).

Column : DB-WAX 15m × 0.320mm × 0.5μm (15m 長 × 0.320mm 内徑 × 0.5μm 膜厚)

Procedure

A. The analysis conditions for gas chromatography are as follows:

1. Adjust the flow rate of the carrier gas (N_2) for column to 2 ml/min.
2. Turn the POWER on.
3. Set AIR gauge reads 40 kPa.
4. Set H_2 gauge reads 55 kPa.
5. Set the injection port temperature to $120^\circ C$.
6. Set the flame ionization detector (FID) to $180^\circ C$
7. Set oven temperature program (column temperature): Initial temperature $60^\circ C$, hold for 1 minutes, then increase by $25^\circ C$ per minute to $110^\circ C$.
9. Injection method: Split.
10. Split ratio: 100:1

B. Qualitative and Quantitative Analysis of Alcohols

1. Inject 0.5 μl for methanol, ethanol, propanol, and butanol, respectively.

Note: To avoid contamination, the syringe should be thoroughly rinsed with **acetone**, followed by **6 to 7** rinses with new sample solution before injecting it.

2. Calibration curve: Make a series of injections of **0.5, 1.0, 1.5, and 2.0 μl** of the provided known alcohols (CH_3OH : C_2H_5OH : C_3H_7OH : C_4H_9OH = 2:1:1:1). The injected actual volume of each species is proportional to the peak area. Therefore, there will be four calibration curves for methanol, ethanol, propanol, and butanol, respectively.

C. Determining the Alcohols and Their Proportions in an Unknown Sample

1. Inject **1.0 μl** of unknown alcohol. Measure the retention time for each of the unknown components and identify them by comparison with the retention time of the known compound.
2. Using each calibration line, estimate the volume composition of your unknown sample.
[e.g., methanol : propanol (vol/vol) = 3 : 1]

D. To shutdown the GC and then close the main valves for the H_2 , AIR and carrier gas (N_2).

儀器設定:

Column type :

Carrier gas :

Carrier gas flow rate :

inject temperature :

Injection method :

Split ratio :

Column temperature :

Detector :

detector temperature :

結果:

(1)定性分析:同一物種，滯留時間(t_r)相同。

(2)定量分析:同一物種，訊號面積與注射體積成正比。

(3)在 GC 層析圖上標明物種，並確認未知物為何種物種。

(4)利用線性迴歸，求未知物中所含各物種的比例。

計算:

混合物比例 甲醇:乙醇:丙醇:丁醇 = 2:1:1:1

v(μl)	0.2	0.4	0.6	0.8	線性方程式，r
甲醇; t _r : min peak area					
v(μl)	0.1	0.2	0.3	0.4	線性方程式，r
乙醇; t _r : min peak area					
丙醇; t _r : min peak area					
丁醇; t _r : min peak area					
unknown no:	t _r : min	t _r : min	經由計算求得 unknown 中物種比例為:		
物 種					
peak area (1.0 μl)					