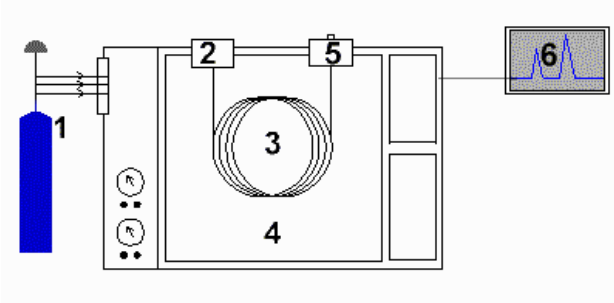
GC Instrumentation
氣相層析儀~儀器簡述與操作

All gas chromatography instruments have the following constituents, all of which are equally important without one of these the GC would cease to work.

1. Provide a constant flow of carrier gas (mobile phase).
2. Permit the introduction of sample vapors into the flowing gas stream.
3. Contain an appropriate length of stationary phase
4. Maintain the column at the appropriate temperature / temperature program.
5. Detect the sample components as they elute from the column.
6. Provide a reasonable signal proportional to the amount of each component.



Gas Supply system

As mentioned earlier the separation in GC, as the name implies, is carried out by a gaseous mobile phase passing through a column. It is essential for the carrier gas supply to the column to consist of a pure oxygen free gas at the appropriate flow rate.

Several gas supplies are usually needed to operate a capillary GC system, namely the carrier gas, hydrogen fuel gas for flame detectors, clean air to sustain the flame combustion, and make up gas if required. All of these gases can be supplied from normal cylinders via the normal cylinder head pressure control valves to reduce the pressure to 5-6 bar.

Whatever the source the various gas supplies must be 'pure'. In the case of the carrier gas it is essential for oxygen, moisture and organic impurities to be absent. Oxygen, in particular, must me absent from the carrier gas to avoid any oxidation of the minute quantity of stationary phase in the capillary column. Any oxidation will result in the loss of column performance. High purity gases should always be used. However it is also essential to include a moisture filter followed by an oxygen filter in the supply carrier gas line.

Flame detectors are ubiquitous in capillary GC, and require auxiliary supplies of hydrogen and air. All auxiliary gas supplies should be cleansed by charcoal filters to remove any traces of organic impurities.

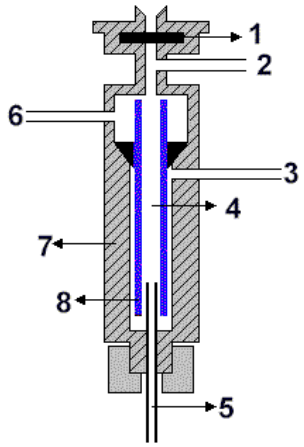
Some recommendations are：

1. Replace gas cylinders whilst still partially full i.e. with 10-15 bar residual pressure.
2. Ensure that the cylinder head fitting is dry and free from any grease, and is in good condition.
3. Use either stainless steel or annealed copper tubing for plumbing. Ensure that all tubing has been washed with solvent to remove grease, etc., and then thoroughly dried. Polymer tubing should not be used as most gases, particularly helium and hydrogen can diffuse through the tube walls. Helium can diffuse out and oxygen can diffuse in!!!
4. Use good quality compression fittings for any connections and always check for leaks.
5. Ensure that the system can be isolated to prevent any ingress of air when cylinders or filters need to be changed. This will require the need for a separate shut-off valve near the supply cylinder.

Sample injection

The sample introduction methods for packed and capillary columns are different because of the sample capacities of these two types of columns. The introduction on to the packed column is usually problem-free because the total sample, which can be introduced with a syringe into an external vaporiser, may also enter the column. This is different with capillary columns, the sample capacity for which is much lower. Therefore a compromise has to be found which depends on the influence of column overload on the resolution and the necessity to introduce large samples in order to attain high signal to noise ratio's for trace components.

The common method used for sample injection into a packed column is direct injection with a hypodermic syringe though a self-sealing silicon rubber septum onto a glass liner within a metal block. The sample is vaporised here and swept into the column.



1) Rubber septum
2) Purge outlet
3) Split outlet
4) Vaporisation chamber
5) Column
6) Carrier gas inlet
7) Heated metal block
8) Glass inner

The block is heated to a temperature sufficiently high to virtually instantly convert the sample into a cloud of vapour. Above is diagram of a split splitless injector, this of particular use in capillary columns where column overload must be avoided. It allows excess sample to be vented out and only a limited /determined amount enter the column. This does however waste significant amounts of sample.

A fixed loop method can also be employed (more common in HPLC); this allows the injection of a fixed volume every time thereby eliminating changes in sample volume and the chance of an overload occurring.

GC Instrumentation
氣相層析儀~儀器簡述與操作 ---2---

Another method is the use of an auto sampler. This device automatically draws up the required amount of the sample from sealed vial and injects it on to the column. Once the run has been completed it injects the next sample and so on. This is used when many samples need to be analyzed.

Chromatographic Oven

The retention of species in a column depends on the temperature of the column. It therefore essential, to control this variable as accurately and precisely as possible. To ensure that there are no temperature gradients in the oven space. Any cold spots in the oven will cause a deterioration of chromatographic performance with peak distortion.

The oven physically is the larges component of a GC machine, and is the central feature to which all other components are linked. Most GC ovens are heated electrically with forced air circulation by a fan, which is mounted at the back. The usual temperature range is from room temperature to 400-500 ^{o}C.

The most important GC oven properties for the potential user are given below:

1. Minimum temperatures**;** the minimum temperature at which the oven can run with accurate control. This is usually 10-20 ^{o}C above ambient.
2. Maximum temperatures**;** the maximum temperature at which the oven can run with accurate control. Values range from 300-500 ^{o}C. Columns are now available for high temperature use, which can be operated up to 450-500 ^{o}C.
3. Accuracy and precision characteristics; a modern oven should be controllable to with in 0.1 ^{o}C of the required temperature and to maintain this value to within 0.1 ^{o}C for the duration of the run. Reproducibility of the programming conditions of the run to run should be within 0.2 ^{o}C.
4. Programme rates; a range of temperature programming rates should be provided, including the ability to change the arte, or to include isothermal periods within the run.
5. Rapid cool-down; this is very important as the cool-down time adds to the overall analysis time.
6. Rapid heat-up; on switching on the column oven it should be capable heating up rapidly.

Chromatographic columns

Open tubular columns (OTC)

Compared with packed columns, OTC offer:

1. Higher resolution
2. Shorter analysis time
3. Greater selectivity
4. Lower sample capacity

Open tubular columns can be classified into three basic types, namely,

Wall coated open tubular (WCOT) –Liquid stationary phase is coated on the inside wall of column. (Figure 2a)

Support coated open tubular (SCOT) – Liquid stationary phase is coated on solid support attached to the inside wall of column (figure 2b)

Porous-layer coated open tubular (PLOT) –Solid stationary phase coated on inside wall of column (figure 2c)

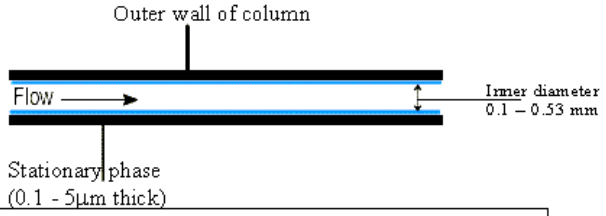


Figure 1 below shows a normal coated open tubular column.

Most open tubular columns are made of fused silica (SiO_2) and coated with polyimide (a plastic capable of withstanding 350^{o}C) for support and protection from atmospheric moisture. Column i.d. are usually 0.10 to 0.53 mm and typical lengths are 15 to 100m. OTC provides higher resolution, shorter analysis times and greater sensitivity than packed columns, but they have lower capacity for samples.

Wall coated columns usually have a 0.1 to 0.5μm think film of stationary phase on the inner wall of the column. Decreasing the thickness of the stationary phase increases resolution, decreases retention time and decreases sample capacity.

Support coated columns (PLOT and SCOT) (figure 2) columns have an increased surface area and therefore handle larger samples than wall coated columns. Support-coated columns are an intermediate between wall coated and packed columns.

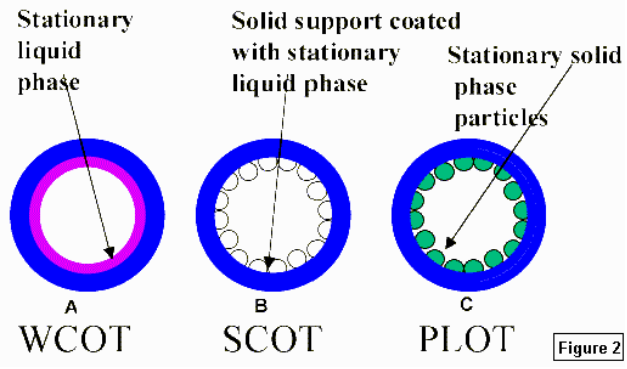


Figure 2

Packed columns

Packed columns contain a fine solid support coated with a non-volatile liquid stationary phase, or the solid itself may be the stationary phase. Although packed columns are not as good as capillary columns, however they are still used. They are useful for preparative separations where a large quantity of stationary phase is required.

These columns are usually made of stainless steel, nickel, or glass and are typically 3-6mm in diameter and 1-5 m in length. The solid support is usually diatomite. For more strongly bound solutes Teflon© is used.

In a packed column, uniform partial size reduces the multiple pathway term in the Van Deemter equation, thereby reducing the plate height and increasing resolution. Small particle size also means that more pressure is required to force the mobile phase through the column.