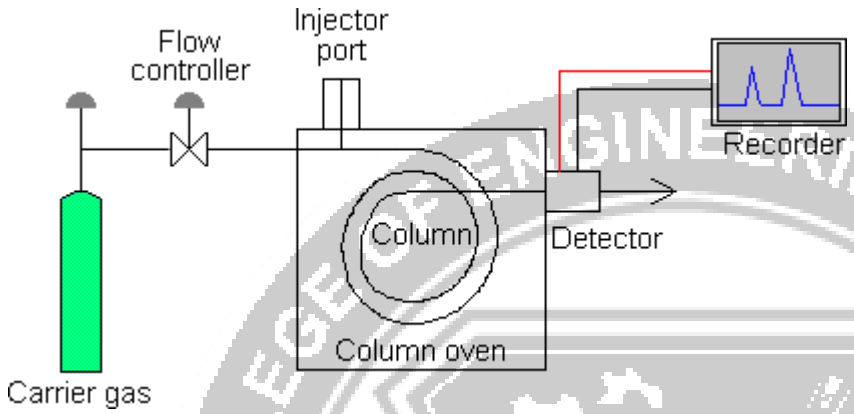
CONSTRUCTIONANDWORKINGOFGASCHROMATOGRAPHY

Introduction



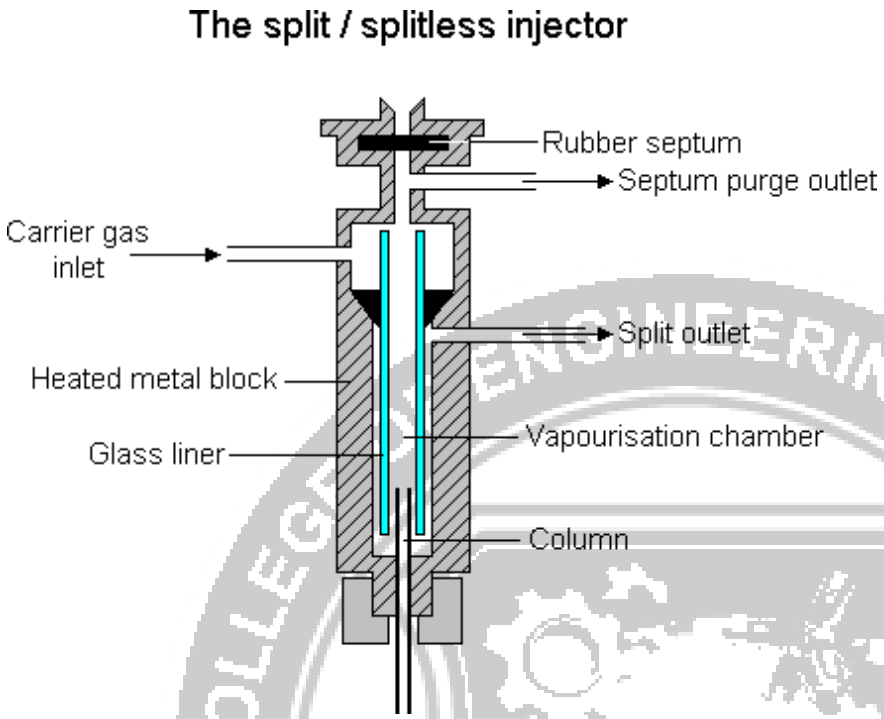
Gas chromatography - specifically gas-liquid chromatography -involves a samplebeingvaporizedandinjectedontotheheadofthechromatographiccolumn.Thesampleistransported through the column by the flow of inert, gaseous mobile phase. The column itselfcontainsaliquid stationaryphasewhich isadsorbedonto thesurfaceofan inertsolid.Instrumentalcomponents

Carriergas

The carrier gas must be chemically inert. Commonly used gases include nitrogen, helium,argon, and carbon dioxide. The choice of carrier gas is often dependant upon the type ofdetector which is used. The carrier gas system also contains a molecular sieveto removewaterandotherimpurities.

Sampleinjectionport

For optimum column efficiency, the sample should not be too large, and should be introducedontothecolumnasa"plug"ofvapour-slowinjectionoflargesamplescausesbandbroadeningandlossofresolution.Themostcommoninjectionmethodiswhereamicrosyringe is used to inject sample through a rubber septum into a flash vapouriser port atthe head of the column. The temperature of the sample port is usually about 50°C higher thanthe boiling point of the least volatile component of the sample. For packed columns, samplesize ranges from tenths of a microliter up to 20 microliters. Capillary columns, on the otherhand, need much less sample, typically around 10^{-3}mL. For capillary GC, split/splitlessinjectionisused.Have alookatthisdiagramofa split/splitlessinjector;



The injector can be used in one of two modes; split or splitless. The injector contains a heatedchamber containing a glass liner into which the sample is injected through the septum. Thecarrier gas enters the chamber and can leave by three routes (when the injector is in splitmode). The sample vapourises toform a mixture of carrier gas, vapourised solventandvapourised solutes. A proportion of this mixture passes onto the column, but most exitsthrough the split outlet. The septum purge outlet prevents septum bleed components fromenteringthecolumn.

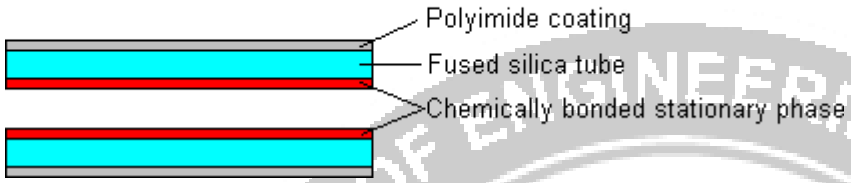
Columns

There are two general types of column, *packed* and *capillary* (also known as *open tubular*).Packed columns contain a finely divided, inert, solid support material (commonly based on*diatomaceous earth*) coated with liquid stationary phase. Most packed columns are 1.5 - 10minlengthandhave aninternaldiameterof2-4mm.

Capillary columns have an internal diameter of a few tenths of a millimeter. They can be oneof two types; *wall-coated open tubular* (WCOT) or *support-coated open tubular* (SCOT).Wall-coated columns consist of a capillary tube whose walls are coated with liquid stationaryphase. In support-coated columns, the inner wall of the capillary is lined with a thin layer ofsupportmaterialsuchasdiatomaceousearth, ontowhichthestationary phasehasbeenadsorbed. SCOT columns are generally less efficient than WCOT columns. Both types ofcapillarycolumnaremore efficientthanpackedcolumns.

In 1979, a new type of WCOT column was devised - the *Fused Silica Open Tubular* (FSOT)column;

Cross section of a Fused Silica Open Tubular Column



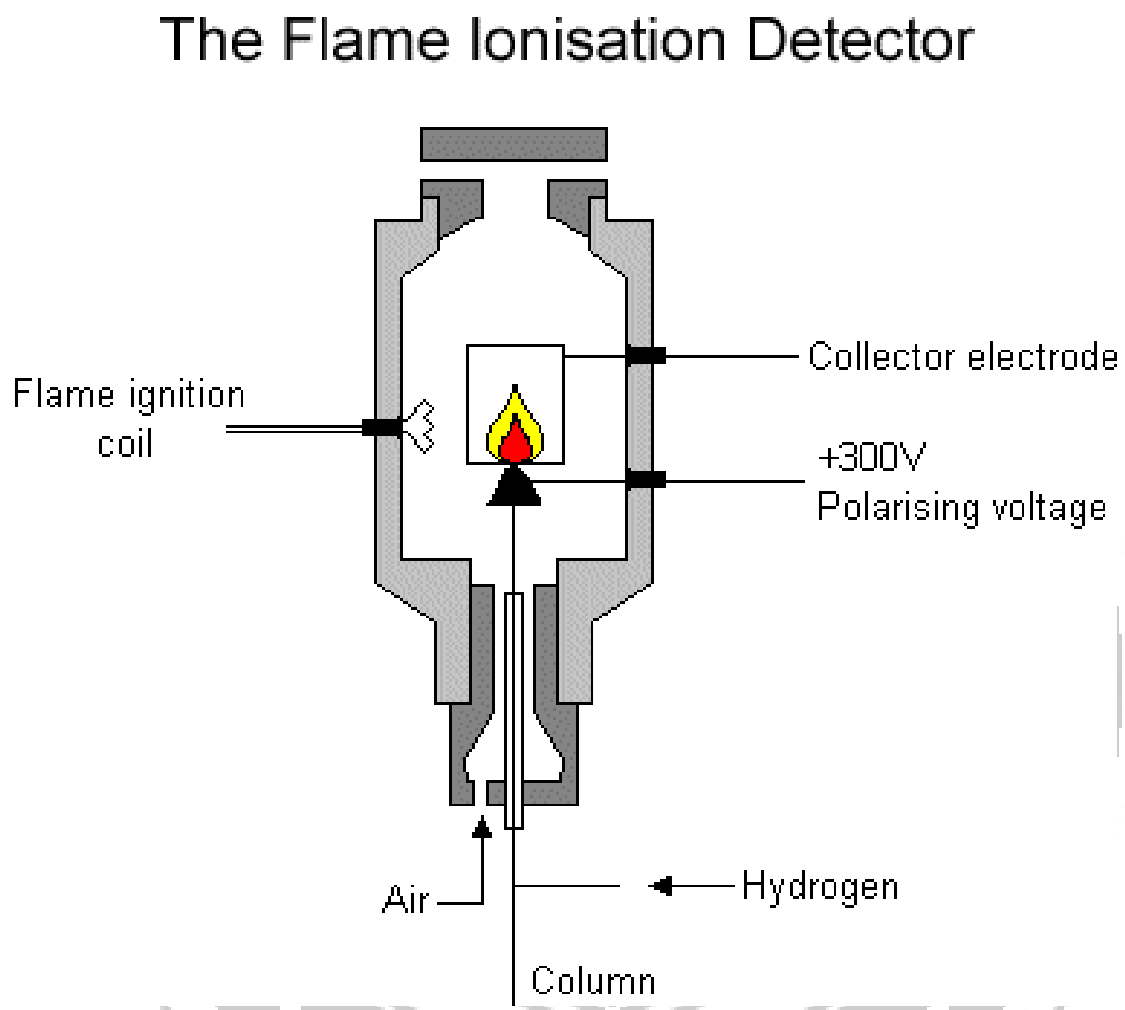
These have much thinner walls than the glass capillary columns, and are given strength by thepolyimide coating. These columns are flexible and can be wound into coils. They have theadvantagesofphysicalstrength,flexibilityandlow reactivity.

Columntemperature

For precise work, column temperature must be controlled to within tenths of a degree. Theoptimum column temperature is dependant upon the boiling point of the sample. As a rule ofthumb, a temperature slightly above the average boiling point of the sample results in anelution time of 2 - 30 minutes. Minimal temperatures give good resolution, but increaseelution times. If a sample has a wide boiling range, then temperature programming can beuseful. The column temperature is increased (either continuously or in steps) as separationproceeds.

Detectors

There are many detectors which can be used in gas chromatography. Different detectors willgive different types of selectivity. A *non-selective* detector responds to all compounds exceptthecarriergas,a*selectivedetector*responds toarangeof compounds with acommonphysicalorchemicalpropertyanda*specificdetector*respondstoasinglechemicalcompound. Detectors can also be grouped into *concentration dependant detectors* and *massflow dependant detectors*. The signal from a concentration dependant detector is related to theconcentration of solute in the detector, and does not usually destroy the sample Dilution ofwith make-up gas will lower the detectors response. Mass flow dependant detectors usuallydestroy the sample, and the signal is related to the rate at which solute molecules enter thedetector.Theresponseofamassflowdependantdetectorisunaffected bymake-up gas.



Theeffluentfromthecolumnismixedwithhydrogenandair,andignited.Organiccompounds burning in the flame produce ions and electrons which can conduct electricitythrough the flame. A large electrical potential is applied at the burner tip, and a collectorelectrode is located above the flame. The current resulting from the pyrolysis of any organiccompounds is measured. FIDs are mass sensitiverather than concentration sensitive; thisgives the advantage that changesin mobile phaseflow rate do not affect the detector'sresponse. The FID is a useful general detector for the analysis of organic compounds; it hashigh sensitivity, a large linear response range, and low noise. It is also robust and easy to use,butunfortunately,itdestroysthesample.