

Gas Chromatography

Chromatography

Term for separation of mixtures based on the partition of substances between two immiscible heterogeneous phases, one stationary, and the other mobile.

Gas Chromatography

The mobile phase is gaseous (or made to be gaseous). It was developed in the 1950s and it is widely used to separate, detect and quantify individual components of nanogram quantities from a volatilized sample in a single step.

Gas-Solid Chromatography

A gas moves over a solid stationary phase. Mainly used for gases.

Gas-Liquid Chromatography

Gas moves over a liquid phase supported by a solid stationary phase. Used for gases and vapors.

Principle of Operation

Nanogram amounts of the gaseous or vaporized components are carried under controlled temperature conditions by an inert carrier gas through a column containing the stationary phase. The components are separated due to differences in vapor pressure, boiling point, absorption, solubility, and chemical bonding into distinct bands of molecules. The fraction zones move at different rates through the column and emerge as separated components.

Components

Carrier Gas

Inert mobile phase to transport the sample through the stationary phase (column).
Need a constant flowrate, so flow controllers and meters are required.
It cannot interfere with the detector.
Examples: He, N₂, Ar, CH₄

Injector System

Is at the beginning of the column and ensures the samples are introduced into the column in a reproducible manner.
Requires precise syringes.
Injection is through a septum, which can be a source of air leaks and contamination from septum material bleed.

Column

This is the most important part, as it does the separation.

Is made of inert material, stainless steel or glass.

Long columns enhance separation.

Small diameter columns enhance resolution of the bands.

Small grain size of the solid support allows larger surface area for holding a thin film of liquid phase enhancing resolution.

The column is located in an oven where the temperature can be carefully controlled.

There are hundreds to choose from.

Detector

Senses the material exiting the column. A good detector is sensitive, has a high signal to noise ratio, has zero drift, is linear over a wide range of concentrations and has consistent response characteristics.

Examples: thermal conductivity, flame ionization, electron capture, photoionization and mass spectrometer.

Output

A chromatogram is obtained which is a plot of the intensity of the detector response versus time. Retention time is used as a presumptive identification of the compound and the detector response (peak area) is proportional to the concentration.

You must know what you are looking for, and you must run standards to know the retention time of the compound of interest and the detector response to that compound.

With proper selection of a carrier gas, injection system, column, detector, temperature and gas flow, you can separate and quantify almost any compound.

The HP 6890 GC/FID is equipped with a general purpose capillary column capable of separating a wide variety of chemicals:

30m x 0.32mm x 0.25 μ m film thickness of 5% phenyl methyl siloxane.

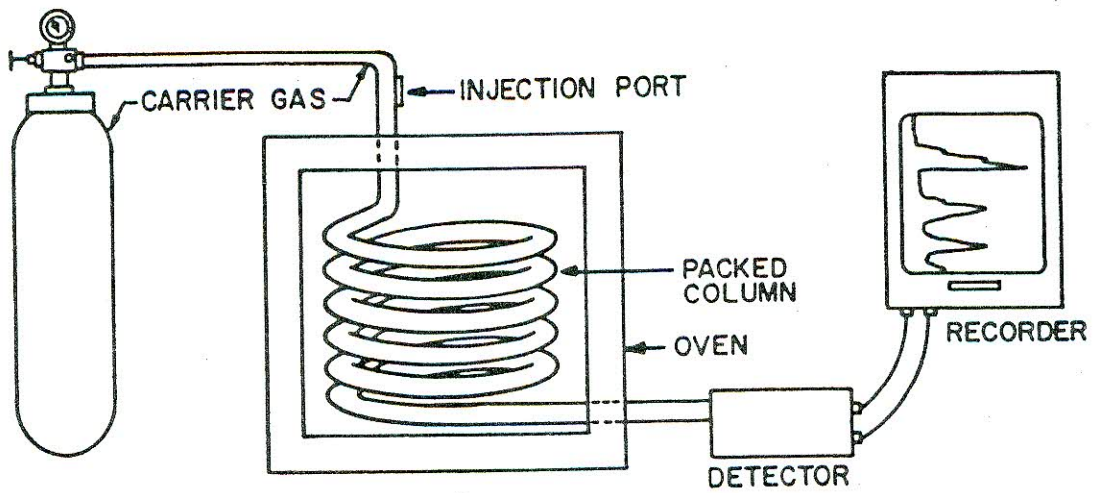


Figure 10-1. Block diagram of a typical gas chromatograph. (From *The Industrial Environment—Its Evaluation & Control*, NIOSH, Washington, D.C., 1973, p. 259.)

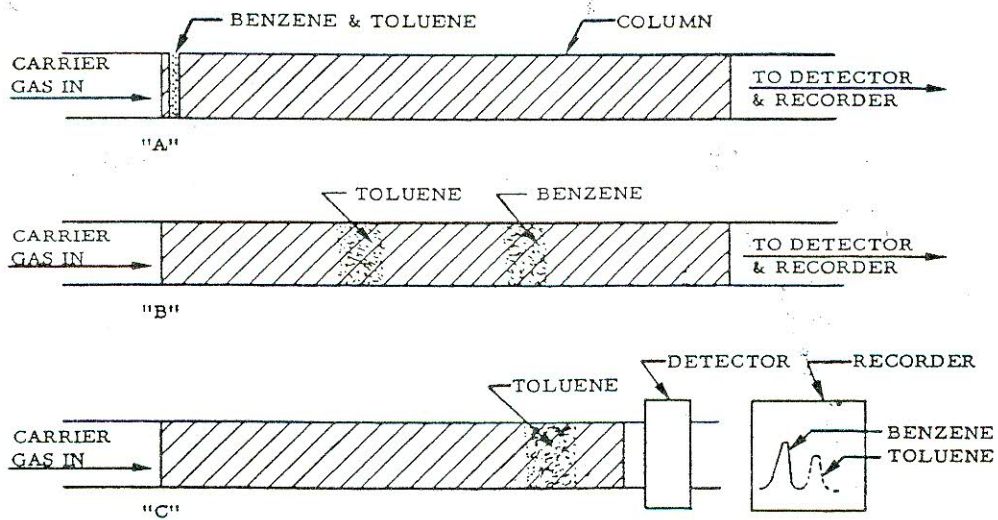


Figure 10-2. Chromatogram/pictorial separation of benzene and toluene. (Courtesy The Foxboro Company.)

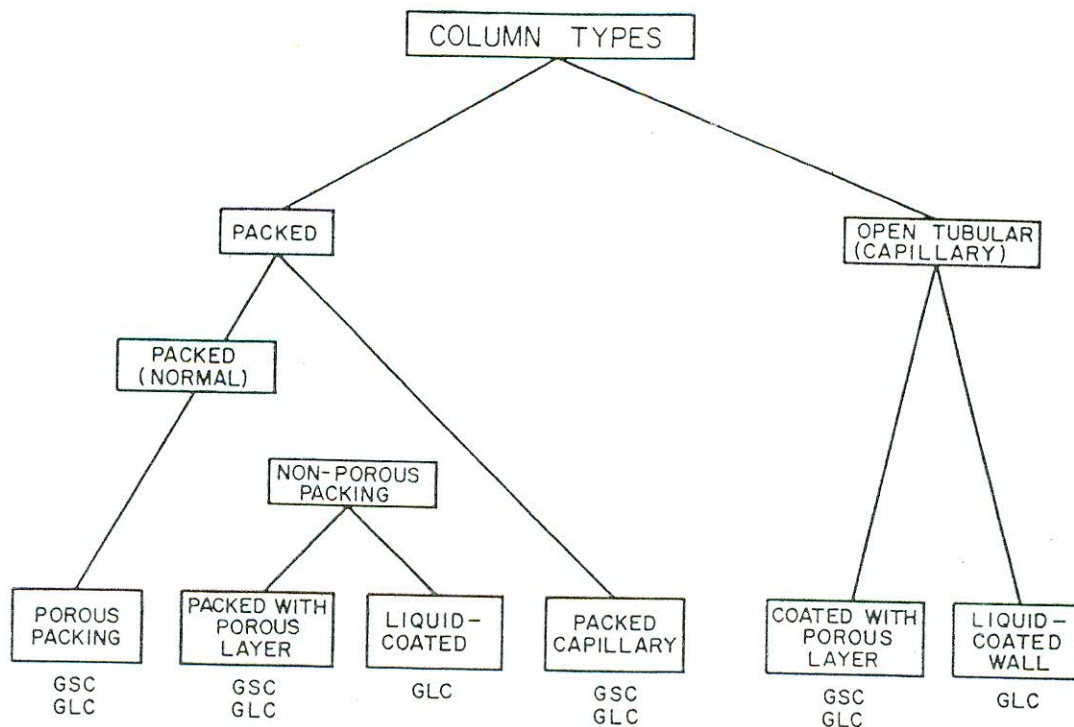
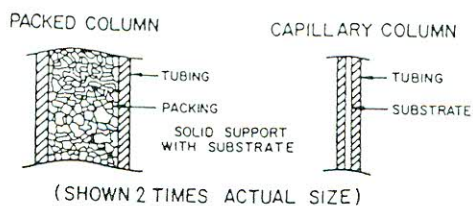


Figure 10-4. Packed and capillary GC columns. (From *The Industrial Environment—Its Evaluation & Control*, NIOSH, Cincinnati, OH, 1973, p. 262.)

TABLE 10-1. Commonly Used Stationary Phases for GC

Applications	
<i>Liquid Phases</i>	
SE-30, OV-1 (methyl silicones)	Hydrocarbons, chlorinated hydrocarbons
OV-1, SE-54 (methyl/phenyl silicones)	PAHs, chlorinated pesticides, hydrocarbons
Carbonax 20M (polyethylene glycol)	Polar compounds such as esters, alcohols
FFAP, SP-1000 (polyethylene glycol terephthalate)	Phenols, volatile acids
HP-5 (diphenyl dimethyl siloxane)	
<i>Solid Phases (Packed Columns Only)</i>	
Chromosorb, Porapak series (styrene/divinylbenzene polymers)	Volatile alcohols, ketones, hydrocarbons, halocarbons (boiling points 30 – 100°C)
Carbon molecular sieves (Carbosphere, Sphero carb, Carbosieve)	C ₁ – C ₅ hydrocarbons
Porous silica (Unibeads, Porasil A)	C ₁ – C ₅ hydrocarbons

