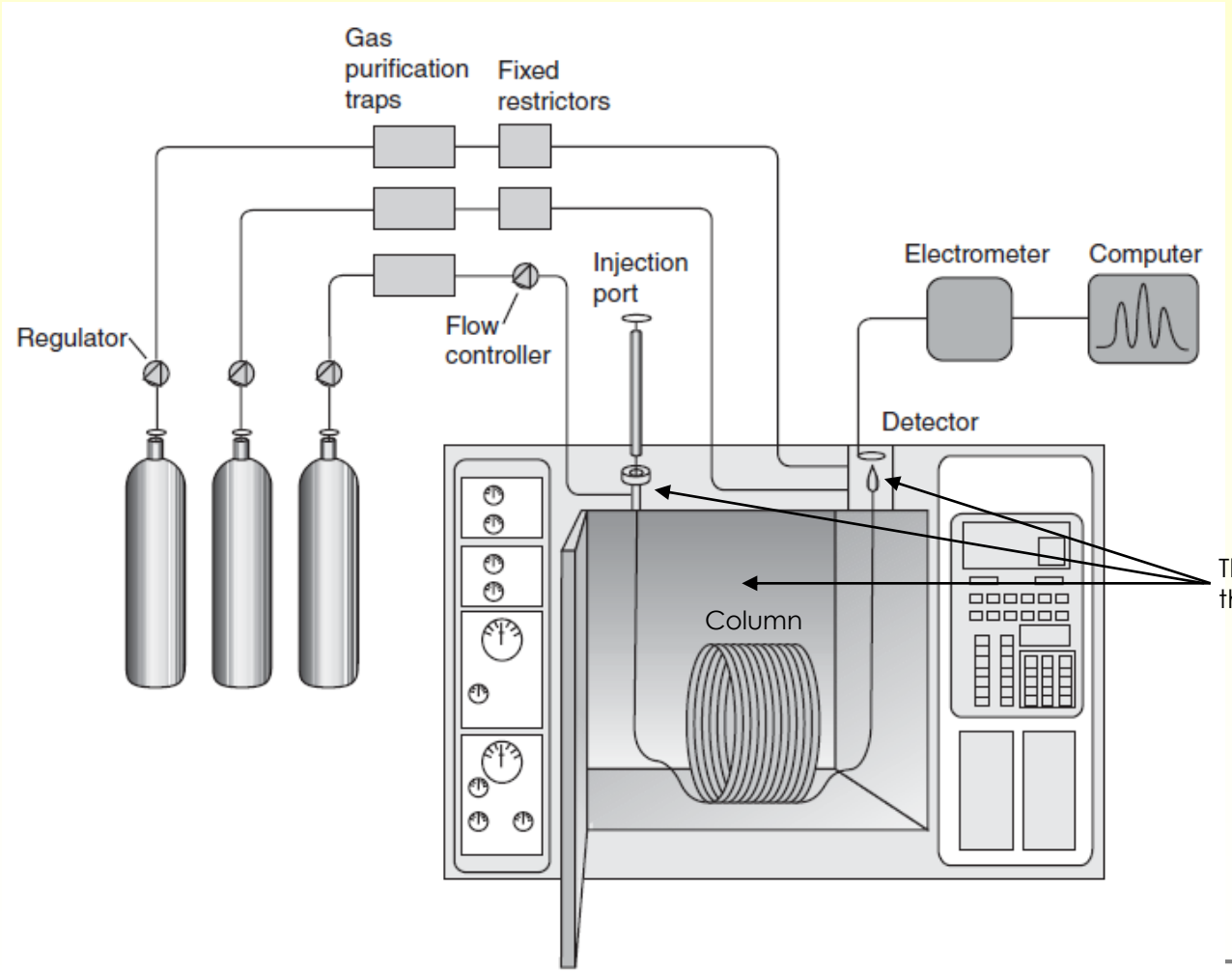
Gas Chomatography

Introduction

In **gas chromatography** (GC) we inject the sample, which may be a gas or a liquid, into an gaseous mobile phase (often called the carrier gas). The mobile phase carries the sample through a packed or capillary column that separates the sample's components based on their ability to partition between the mobile phase and the stationary phase.

Gas chromatograph



Carrier gas in GC

The purpose – to move analyte molecules through the column

Must be inert and pure to avoid any interactions in the column

The most common mobile phases in GC are He, N_2 and H_2

Carrier rate depends on the column and is usually 1-25 mL/min for capillary and 25-150 mL/min for packed columns.

Sample introduction in GC

Cool on-column – for thermally labile compounds

Split/splitless – classic for capillary columns

Programmable temperature vaporization – provides maximum flexibility

Only gaseous and liquid samples can be directly injected into GC

All solid and dirty liquid samples require sample preparation

Split injection

Only small part of injected sample (1:1000-1:10) reaches the column

Other part of sample is sent to split vent

Very fast injection > narrow peaks

Required for analysis of high concentrations (to avoid detector overload)

Split injection animation

Open “Split Injection” video

Task 1

One microliter of vodka sample having concentration of methanol 125 mg/L was injected to gas chromatograph in 1:50 split mode. How many nanograms of methanol reached the column?

$$m = \frac{V \times C}{S}$$

Where:

m – mass of analyte reached the column, ng

V – sample volume injected, µL;

C - analyte concentration, ng/µL;

S – split factor (50 in our case)

Task 2

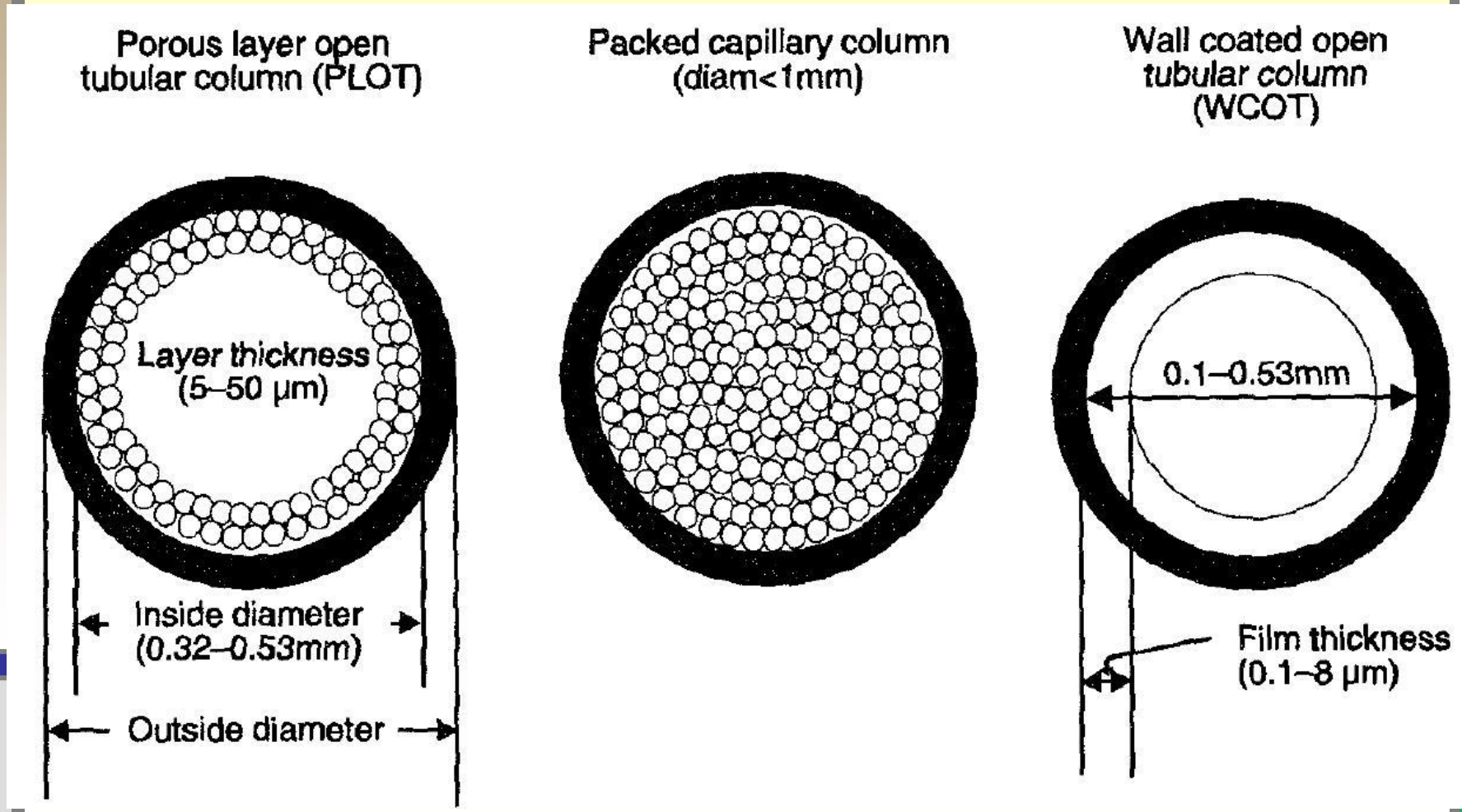
One microliter of naphthalene solution (10 mg/L) in methanol was injected into inlet of gas chromatograph. What volume of gas will be formed in the inlet at temperature 240 °C and pressure 0.49 bar (relative to ambient). Can this sample be injected into a liner having length 68.5 mm and internal diameter 4 mm (vapor volume must be lower than liner volume)?

Absolute Pressure = Ambient Pressure (bar) + 0.49 bar

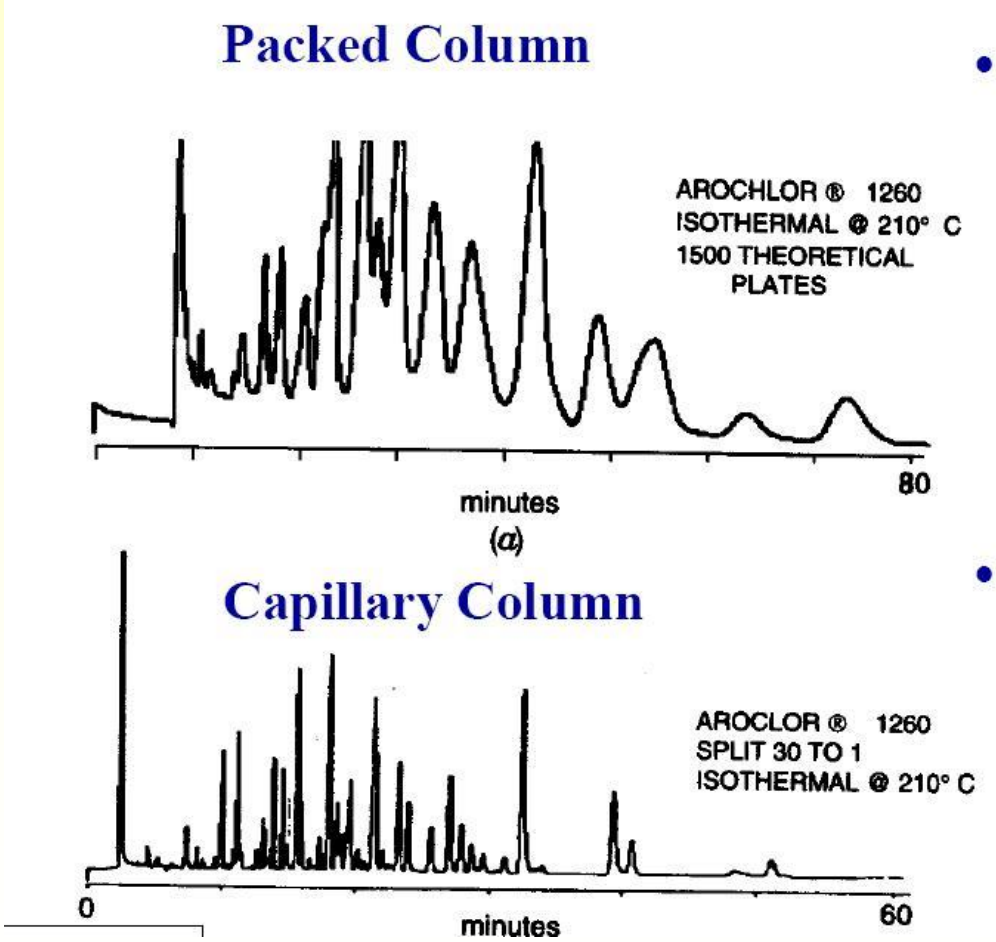
Main formula:

$$p\,V = \frac{m}{M}\,R\,T$$

Columns in GC

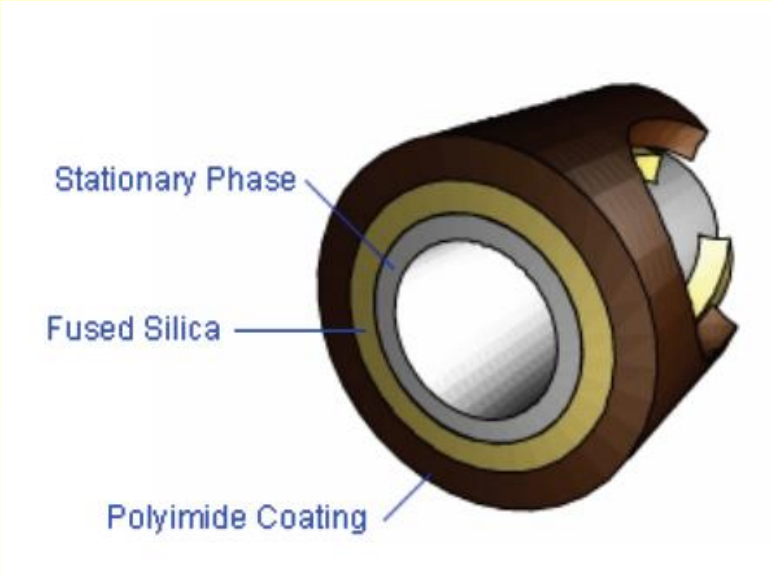


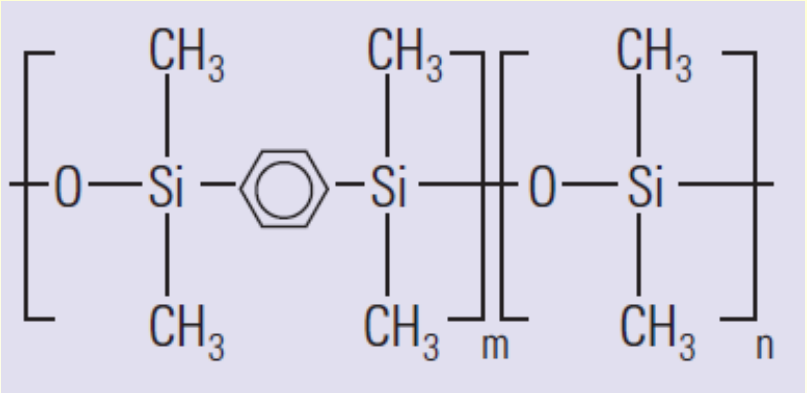
Packed vs capillary columns



- Packed Column
  - Lower resolution
  - Fewer peaks (16)
  - Fewer plates
- Capillary Column
  - Small sample needed
  - Better resolution
  - More peaks
  - Faster Analysis

Capillary columns in GC





Stationary phases:

Non-polar to polar polymers

Polydimethylsiloxane and polyethyleneglycol are mostly used non-polar and polar stationary phases, respectively

Aromatic and other functional groups are implemented into polymeric composition to change polarity of the phase

Main GC parameters

Injection

Mode (split or splitless)

Split ratio (for split mode)

Temperature

Column

Mode (constant flow or constant pressure)

Flow or pressure

Compound retention in GC

Physical properties of analyte (boiling point, molecular weight, diffusion coefficient, polarity)

Column temperature (higher temperature = lower retention)

Carrier flow rate (higher flow rate = lower retention)

Polarity of stationary phase (polar stationary phase stronger retains polar compounds)

If peaks are not separated

Lower temperature

Decrease flow rate

Change stationary phase

Increase column length

If compounds are not retained

Use thicker stationary phase

Use PLOT or packed column

Separate at cryogenic temperatures (use liquid N_2 or CO_2)

Main detectors in GC

Thermal conductivity (TCD)

Flame ionization (FID)

Nitrogen phosphorus (NPD)

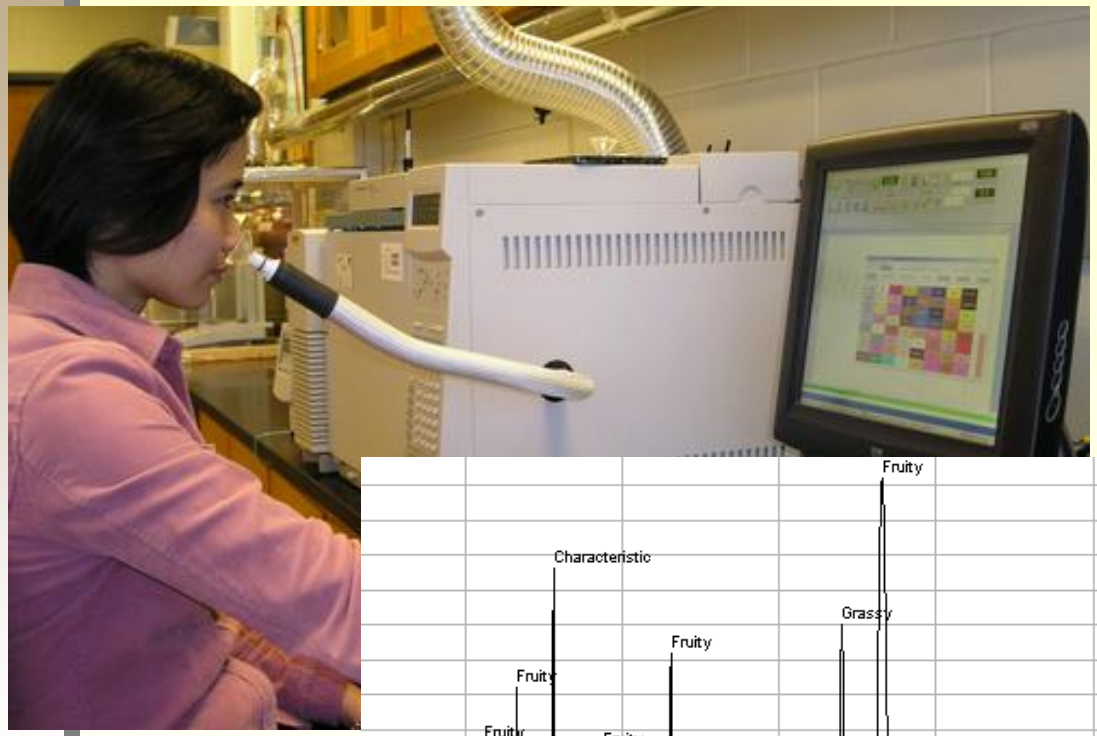
Electron capture (ECD)

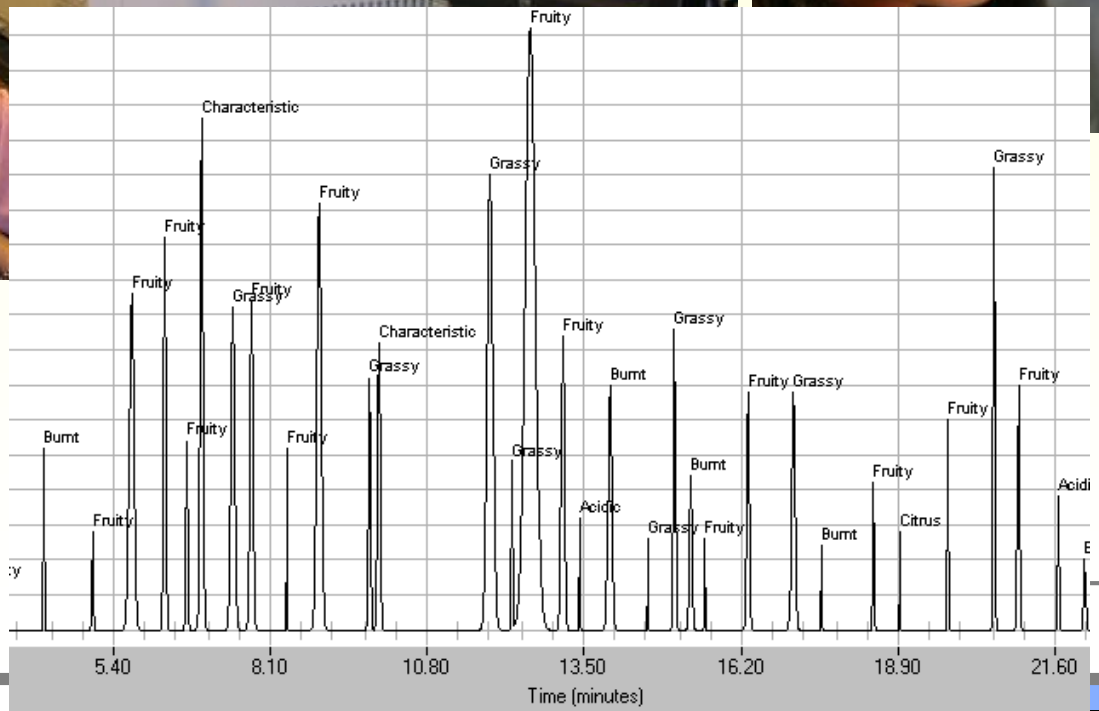
Photo ionization (PID)

Infrared (IR)

Mass spectrometric (MSD)

Olfactory detector





Characterizing odor caused by separated chemicals from one sample

Odor Character

Hedonic tone

Odor Intensity

Task 3

Twenty microliters of air were injected into GC inlet in splitless mode. Expected benzene concentration is 5 ppb (v/v). Will benzene be detected on chromatogram if detector (MS) sensitivity to benzene in 5 pg at signal to noise ratio 5:1?