GAS CHROMATOGRAPHY

Practical Environmental Analysis (EN517)

Practical part

GAS CHROMATOGRAPHY

- In a gas chromatographic system, the sample is volatilized by rapid exposure to a zone kept at relatively high temperature (200-300°C) and mixed with a stream of carrier gas (Ar, He).
- The resulting gaseous mixture enters the separation section (chromatographic column). Upon their displacement through the column, analyte molecules are partitioned between the gas carrier stream (mobile phase) and the column (stationary phase).
- A carrier gas is responsible for carrying the sample molecules into the column and finally to the detector. Carrier gas must be: inert with the stationary phase, of high purity 99.999 %.
- As the compounds are separated, they elute from the column and enter a detector. The detector is capable of creating an electronic signal whenever the presence of a compound is detected; the greater the concentration in the sample, the bigger the signal. The signal is then processed by a computer. The time from when the injection is made (time zero) to when elution occurs is referred to as the retention time (RT).

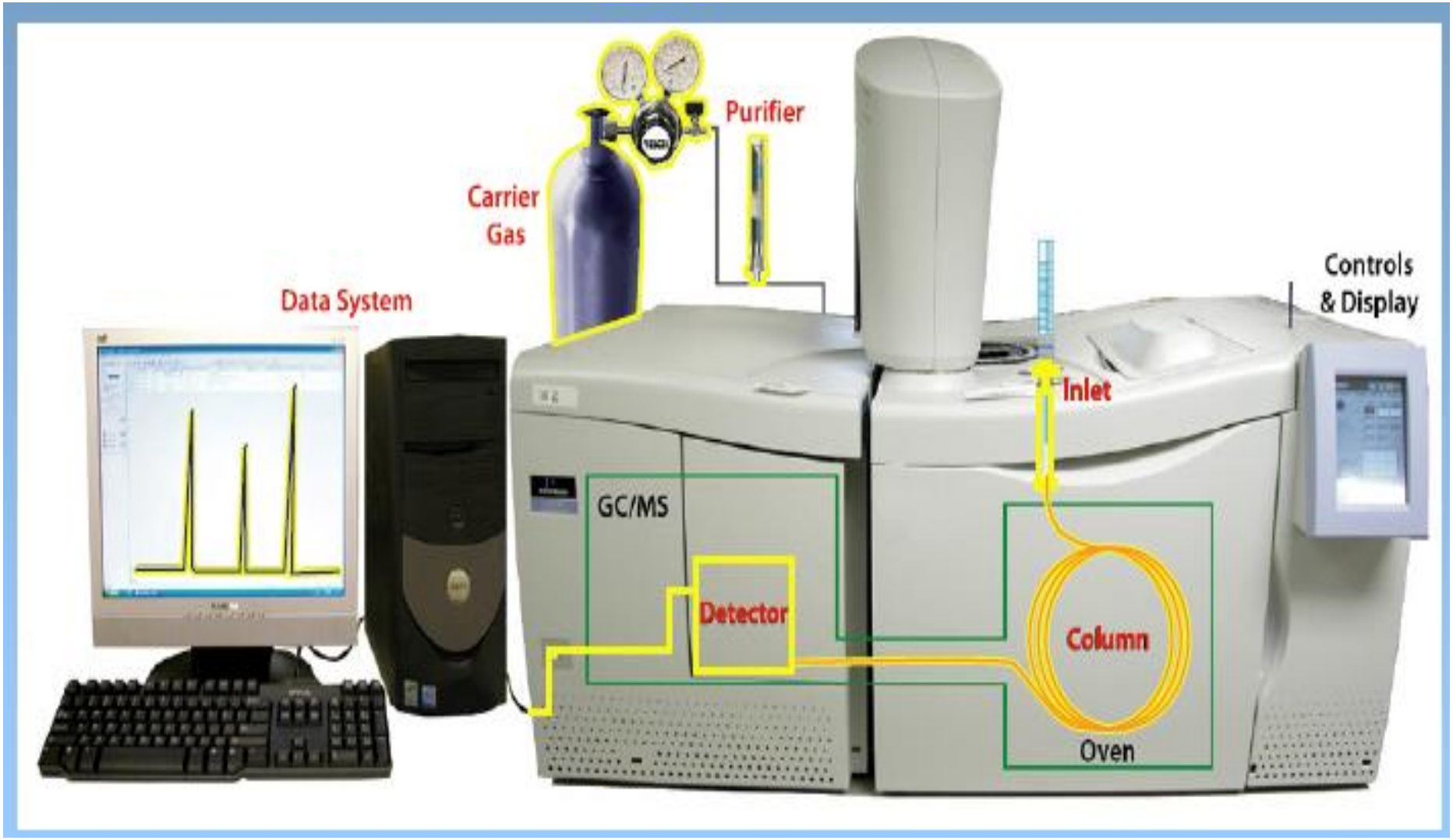
COMPOUNDS AMENABLE TO GAS CHROMATOGRAPHY

- Should be thermally stable
- Should be un-reactive and non-absorptive to chromatographic system
- Should be volatile at temperatures below 350-400°C

SAMPLE PREPARATION FOR GC ANALYSIS

- Sample preparation for GC analysis involves techniques (Distillation, Extraction) which preferentially isolate volatile and semi-volatile substances and prevent the presence of ionic or high molecular weight species in the mixture to be injected into the GC.
- The resulting extracts or distillates are volatile mixtures suitable to GC or GC-MS analysis.

COMPONENTS OF A GAS CHROMATOGRAPH



GC INJECTION TECHNIQUES

- Split Injection
- split-less Injection

SPLIT INJECTOR

- In the split injection mode, sample enters the injection port and is vaporized rapidly.
- Vaporized sample is mixed with a carrier gas (diluted).
- Finally, a large part of the diluted vaporized sample is split away from the Colum, while a small part will enter the column.
- This mode of injection is used for analysis of samples of high analyte concentrations.

SPLIT-LESS INJECTOR

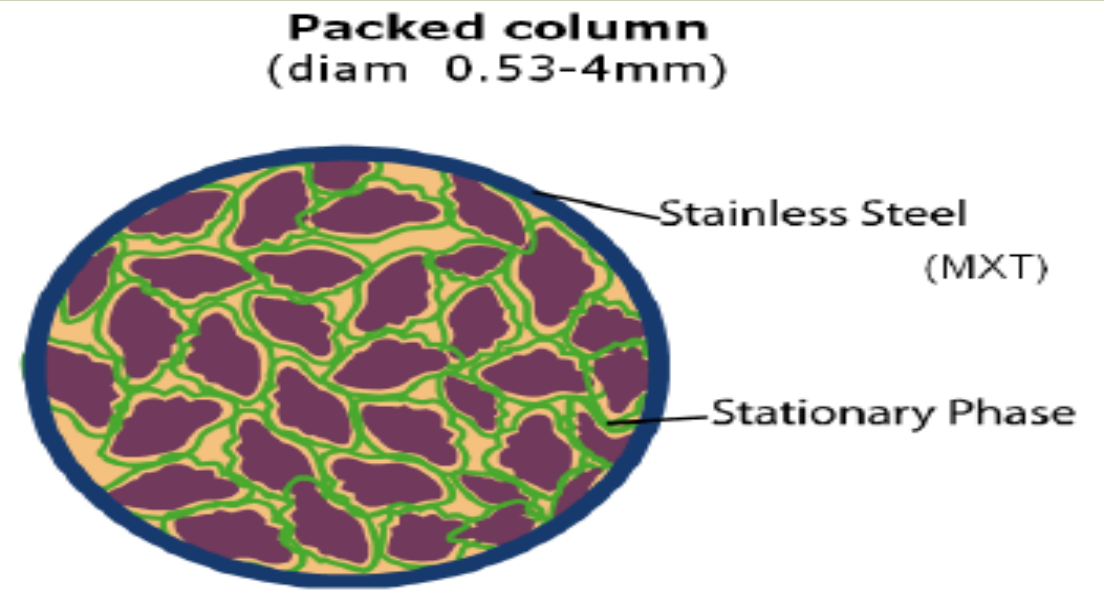
- Is the split-less injection mode, sample enter the injection port and is vaporized rapidly.
- Vaporized sample is mixed with a carrier gas (diluted).
- Finally, all the diluted vaporized sample enter the column.
- This mode of injection is used for analysis of samples of trace analyte concentrations (residue analysis).

GC-COLUMN

1) PACKED GC COLUMNS
2) OPEN TUBULAR/CAPILLARY GC COLUMNS

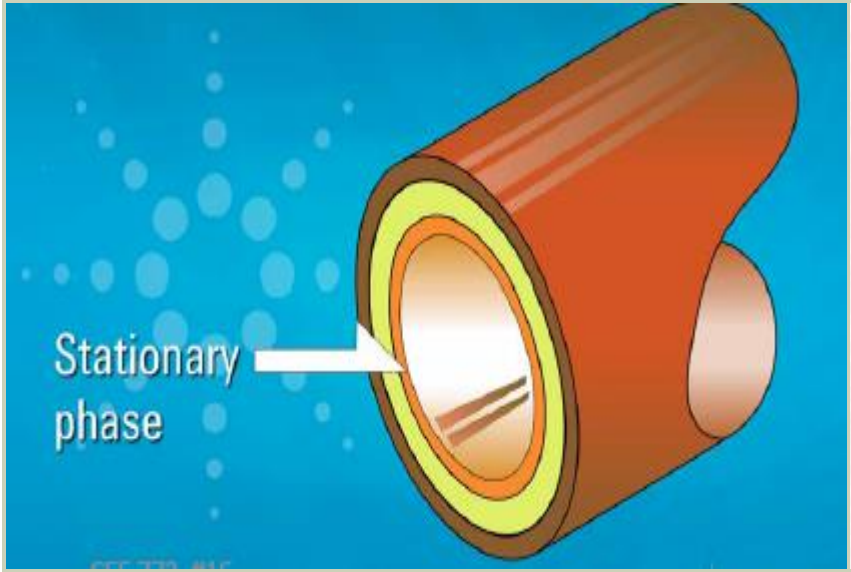
PACKED GC COLUMNS

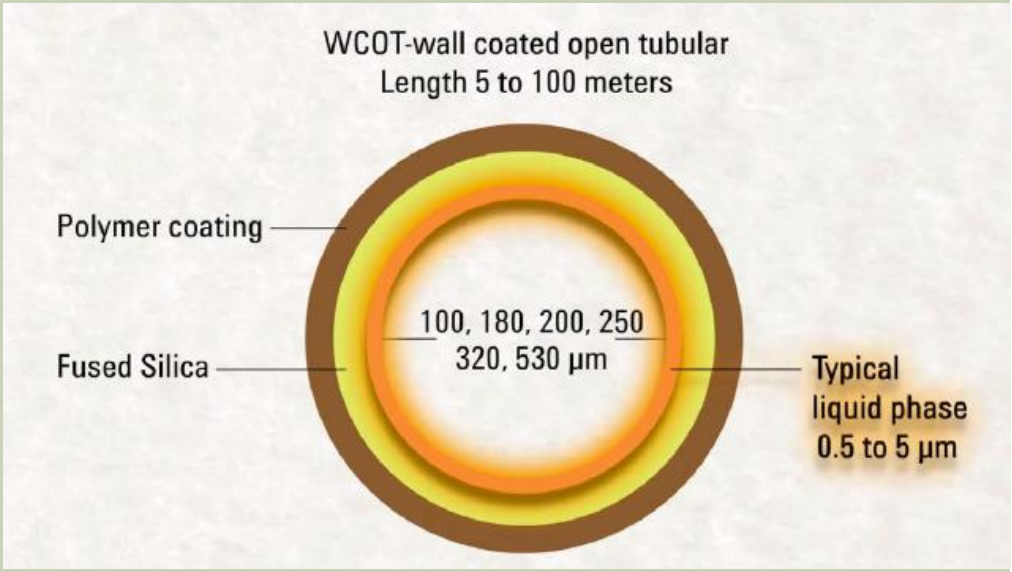
- Less ubiquitous application: fixed gas analysis
- Lower column efficiency than that of capillary columns
- Larger sample capacity
- The efficiency of a gas chromatographic column increases rapidly with decreasing particle diameter of the packing.



OPEN TUBULAR/CAPILLARY GC COLUMNS

- Most widely used
- High column efficiency (large number of theoretical plates due to long column length, up to 100 m)
- Small sample capacity
- Fused silica (pure form of glass) that is very inert but fragile
- Polyamide--provides great mechanical strength and flexibility





STATIONARY PHASE

- In gas-liquid chromatography, the stationary phase is a liquid which is immobilized or adsorbed on a solid support material such as silica particles
- stationary phase for capillary GC column must be low volatility, thermo stability and chemical inertness (non-reactive to both solutes and carrier gas)
- The commonly used stationary phase for capillary GC column is poly dimethyl siloxane[PDMS]. The separation, in this case, will depend upon interactions with nonpolar part of the analytes.
- The PDMS stationary phases can be more polar by adding phenyl, cyano, or trifluoro functional groups each of these stationary phases.
- In gas-solid chromatography, same material is used as both the stationary phase and support material. The common adsorbents used include alumina..

GC DETECTION SYSTEMS/DETECTORS

1) FLAME IONIZATION DETECTOR (FID)

2) MASS SPECTROMETRY

GAS CHROMATOGRAPHY MASS SPECTROMETRY (GC/MS)

- Gas chromatography mass spectrometry(GC/MS)is an instrumental technique, comprising a gas chromatograph(GC)coupled to a mass spectrometer(MS), by which complex mixtures of chemicals may be separated, identified and quantified.
- The GC separates mixes of chemicals into individual components
- The MS fragments the chemicals into unique patterns or spectra.
- As the compounds are separated, they elute from the column and enter a detector. The detector is capable of creating an electronic signal whenever the presence of a compound is detected; the greater the concentration in the sample, the bigger the signal. The signal is then processed by a computer. The time from when the injection is made (time zero) to when elution occurs is referred to as the retention time (RT).

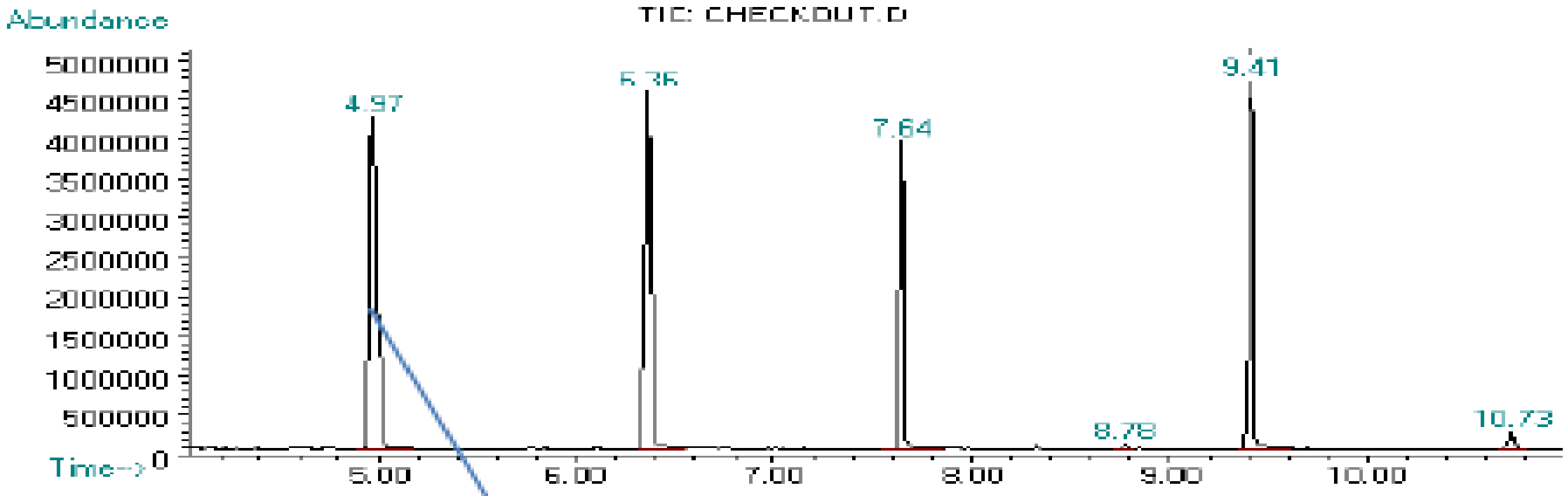


Figure 1: Chromatogram generated by a GC.

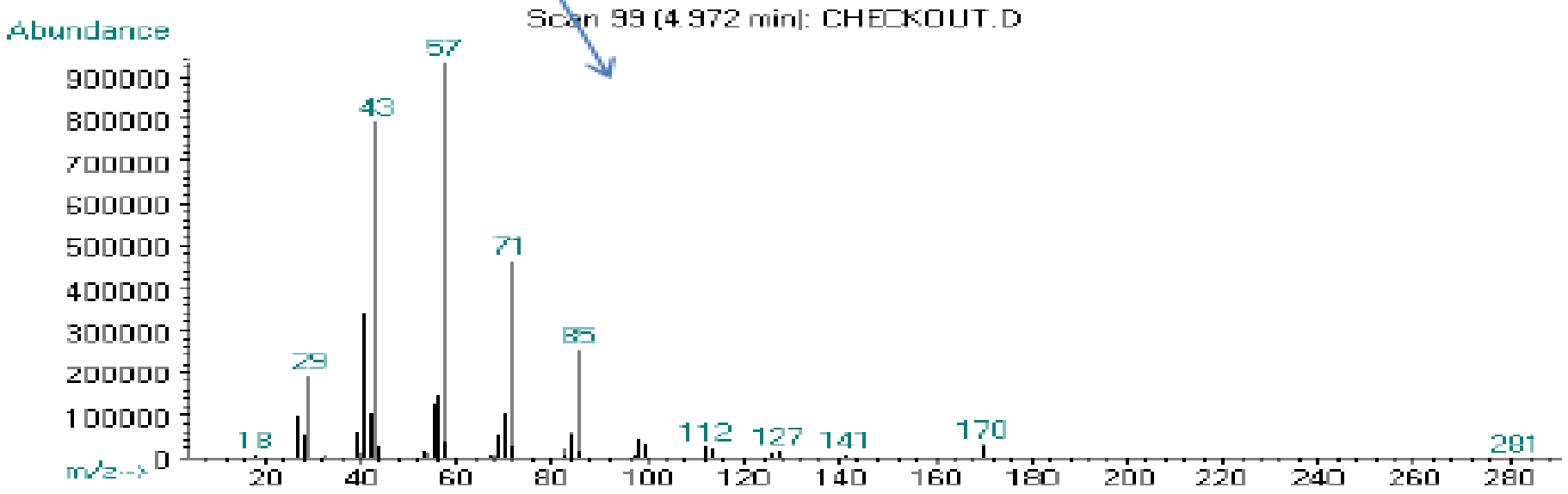


Figure 2: Mass-spectrum generated by an MS.