



Gas Chromatography - Mass Spectrometry (GC-MS)

- Separation of complex mixtures
- Molecular structure identification
- Ultra-low concentrations
- On-site sampling

Gas chromatography coupled to mass spectrometry is a versatile tool to separate, quantify and identify unknown (volatile) organic compounds and permanent gases. By combining sensitivity and a high resolving power, complex mixtures can be analyzed. The information obtained can be used for detection of impurities, contamination control and improvement of, for example, semiconductor manufacturing processes.

Principle of Gas Chromatography – Mass Spectrometry

Gas Chromatography is used to separate volatile compounds in a mixture. The separated compounds can be identified and quantified.

To achieve the identification of different compounds, three steps can be distinguished in a GC-MS system:

Injection

Depending on the sample (gas, liquid or solid), compounds in a mixture need to be volatilized or extracted from the matrix by one of the injection techniques listed in table 1.

Separation

After injection of a mixture, separation is achieved in the capillary column. This column is coated with a fluid or a solid support, the stationary phase. An inert gas, also called the mobile phase, is flowing through the column. Depending on the phase equilibrium between the stationary and mobile phase, compounds travel with different velocities through the column. The mixture becomes separated, and as a result, individual compounds reach the detector with a different retention time. By choosing a column, which separates on boiling point, polarity, size or stereochemistry, a wide range of compounds can be separated.

Detection

Many different detectors (table 2) can be used for detection of the separated compounds. The mass spectrometer (figure 1) combines a high sensitivity with the unique property of being able to determine the molecular composition. Below, only the mass spectrometer will be discussed in more detail. The other detectors are dedicated tools to analyse specific compounds (see also table 2).

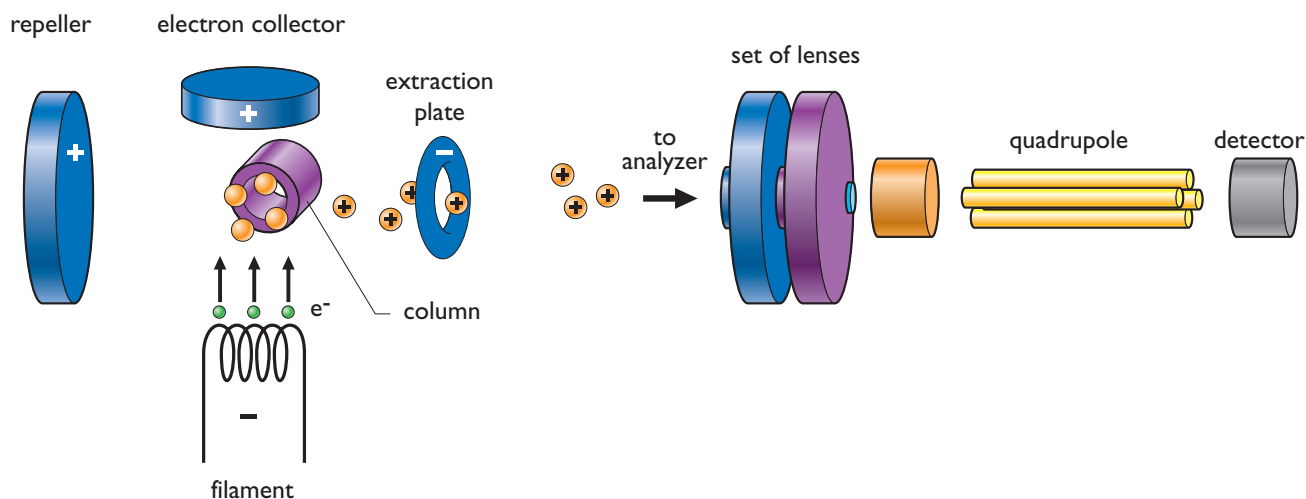
Table 1: Injection techniques and their applications.

Injection Technique	Used for
Split	High concentration samples (liquid/gas) <i>Only small amounts of sample required</i>
Split-less	Trace analysis (liquid/gas)
Thermal Desorption	Trace analysis (gases) and solid samples <i>Desorption of volatile compounds in a solid matrix</i>
Headspace	Volatile compounds in difficult matrices <i>For collection of vapors above dirty samples</i>
Solid Phase Micro Extraction (SPME)	Volatile compounds in difficult matrices with pre-concentration

Table 2: Detection techniques and their applications.

Detector	Used for
Mass spectrometer (MS)	Identification of unknown compounds
Flame Ionization Detector (FID)	Compounds containing Carbon
Thermal Conductivity Detector (TCD)	Universal detection for gases without Carbon
Nitrogen Phosphorous Detector (NPD)	Selective detection of Nitrogen and Phosphor containing compounds
Electron Capture Detector (ECD)	Selective detection of halogen containing compounds
Atomic Emission Detector (AED)	Selective detection of elemental composition

Fig. 1: Schematical drawing of an electron impact ionization source and a quadrupole mass spectrometer. The compounds to be analyzed are represented by the orange spheres.



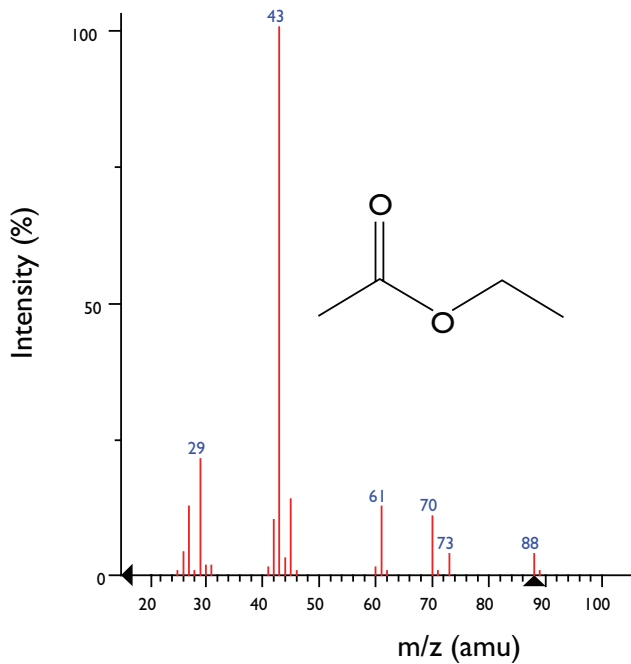


Fig. 2: Mass spectrum depicting the characteristic fragmentation pattern of ethyl acetate ($C_4H_8O_2$) with at $M_w=88.05$ g/mol the molecular (non-fragmented) ion.

Mass spectroscopy

Compounds enter the ion source and are ionized and fragmented by using a high-energy electron bombardment. The ions are extracted from the source with an electric field and fed into the mass analyzer (see figure 1).

By applying electric fields, ions with a certain mass to charge ratio can reach the electron multiplier. The fragmentation pattern measured is characteristic for each molecule, making identification possible (figure 2).

Example 1: Process gases

It is not always possible to bring the process that needs to be analyzed to the laboratory. If, for example, gases used in a semi-conductor process should be analyzed, it is important that the process in which they are used is not interrupted. So-called adsorption tubes (see frontpage image) enable on-site sample collection. In that way, in-situ studies with increased sensitivity are possible in exhaust pipes and process lines. By trapping a large volume of gas (typically several liters), detection limits in the ppt-range can be obtained.

Example 2: Cleanroom air

The presence of airborne molecular contamination (AMC) in cleanroom air can have a negative effect on the achieved yield. To assess the level of AMC in a cleanroom several samples are taken using adsorption tubes and sent to the laboratory for analysis. At the laboratory, the adsorption tubes are desorbed at elevated temperature and the organic contamination present is analyzed using GC-MS (figure 3). Typically, over 100 different compounds are detected, including compounds like cleanroom solvents (acetone, ethyl acetate etc.) and large hydrocarbons. Quantification of the obtained data yields the exact composition of the gas/cleanroom air.

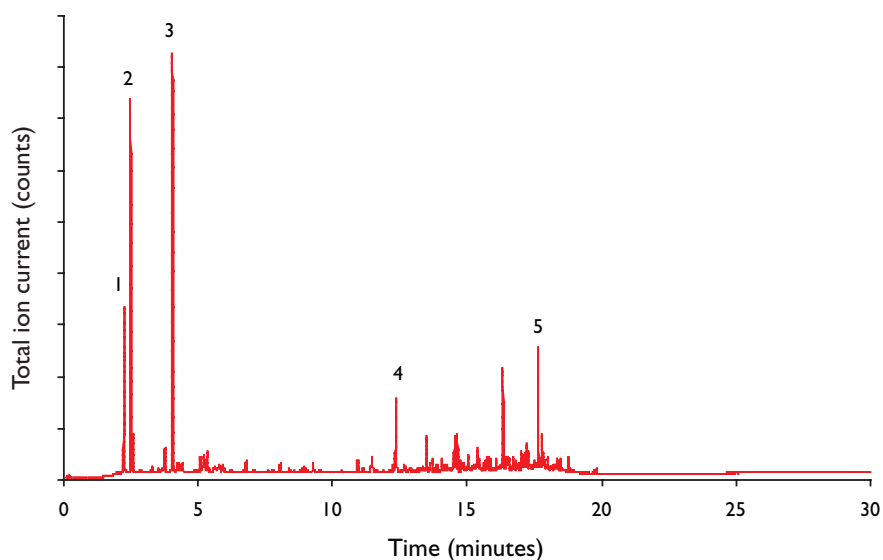


Fig. 3: Chromatogram of cleanroom air. Some of the characteristic peaks are indicated: 1= ethanol, 2= acetone, 3= ethylacetate, 4= cyclohexanone, 5= dodecane.



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Characteristics

Information obtained

- Molecular structure information, molecular composition
- Composition of complex mixtures
- Both qualitative and quantitative

Sample type

- Gases, liquids, solids

Analytical Range

- Ultra-trace (ppt) to main components (%)

Sample size (typical)

- Liquids/solids - milligrams
 - Gases - (milli)liters
- (depending on the required sensitivity)

Accuracy

- < 5% relative

Precision

- < 3% relative



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