GCMS: Quick Guide to the Basics

GC-MS Basics

The purpose of coupling GC with MS is to provide identification of an analyte of interest without requiring further analysis. GC can separate many volatile and semi-volatile compounds but not always selectively detect the constituents. The MS can selectively detect many compounds (by fragmentation pattern) but not always separate them. This is the basis for the operation and use of a GC-MS. In most cases, parallel properties (retention time, isotope ratios) are required to consider the results confirmatory.

GC-MS…How can that work?

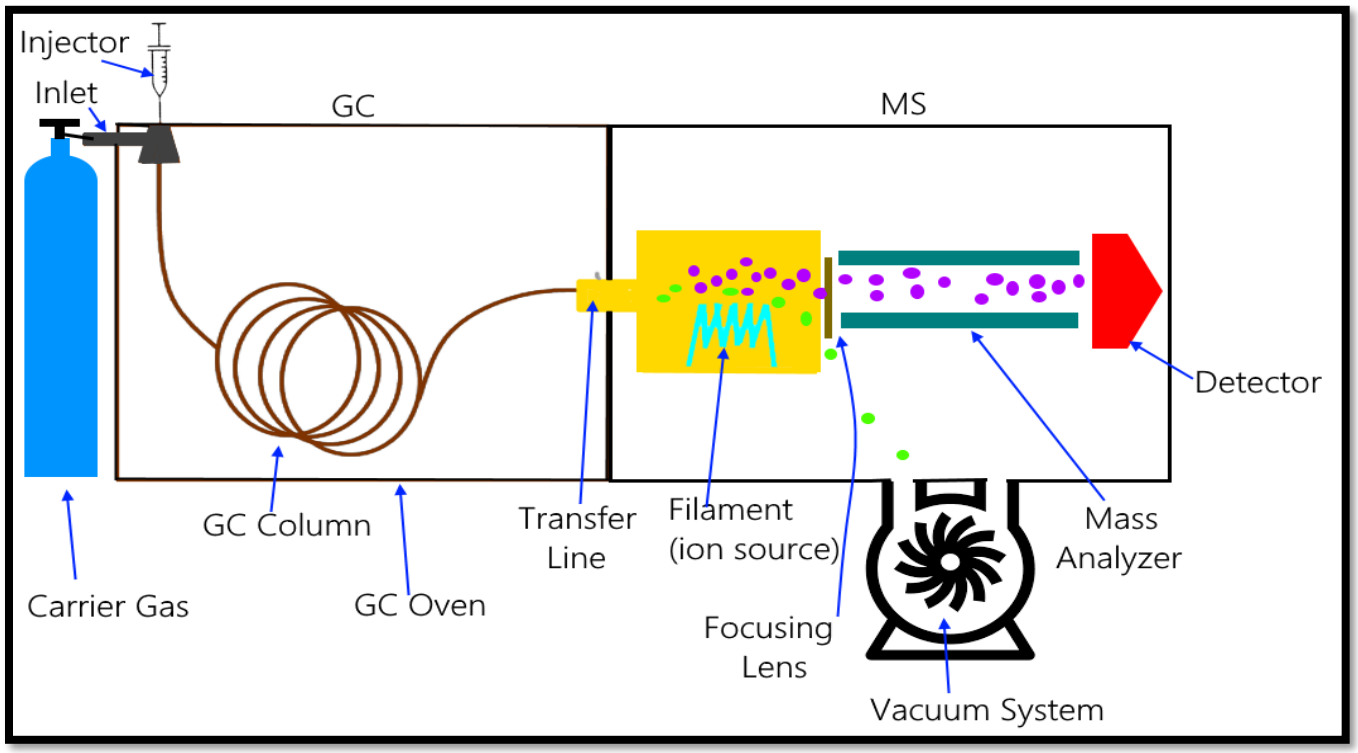
A major concern coupling a GC to a MS is the significant differences in pressure-the GC gas exiting the system is around one atmosphere (760 torr) whereas the MS operates at a vacuum of around $10e^{-6}$ torr. . This issue was solved in capillary GC columns by having the GC gas flow kept low (<4 ml/min) and the pumping speed of the MS vacuum system is high (~20L/sec) then all of the GS effluent can be passed into the MS. With this setup, the capillary GC column directly inserted into the ion source.

GC-MS Instrument Principles

The sample is injected manually or by automation. As the effluent are separated in the GC column into 'individual compounds' they elute from the GC. The compounds enter the MS, and the electron ionization (EI) source generate ions. The generation of ions, results from using a stream of electrons ionizing the molecules and causing them to fragment. The mass of the fragment divided by the charge is the mass charge ratio (m/z). For a GC-MS, using an EI source the charge is usually +1, and m/z ratio represents the molecular weight of the fragment. A group of four electromagnets, a quadrupole, focuses each fragment through a slit into the detector. These quadrupoles are tuned by a computer to direct only certain fragments (please see How Quads Work). The computer has the quadrupoles, which cycle these fragments one at a time (scan) until the range of m/z is completed. This produces the mass spectrum; signal intensity (relative abundance) versus m/z ratios (approximate molecular weight). The knowledge that most

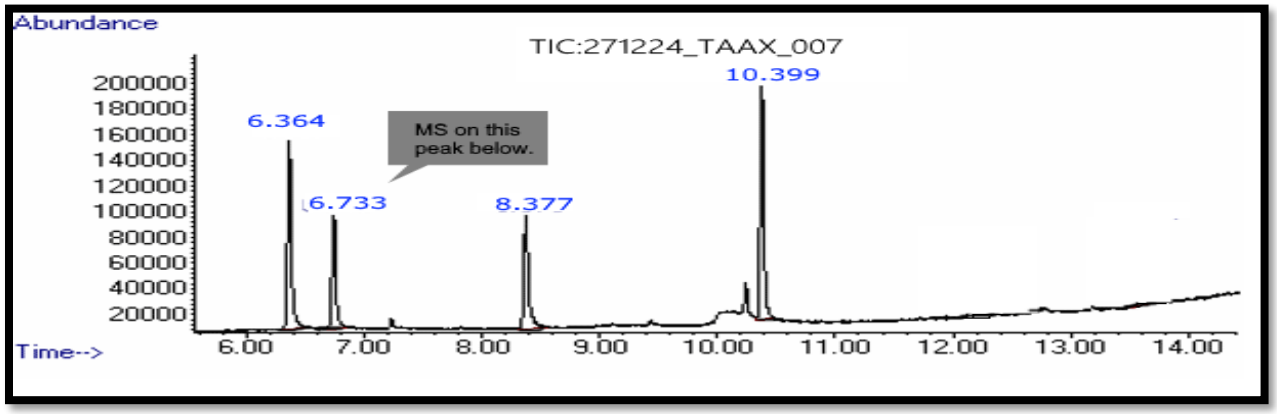
compound (enantiomers excluded) have a unique fragmentation 'fingerprint' and software is readily available to provide a library of spectra for known compounds and comparing them to the fragmentation in question. It is easy to see the power behind the GC-MS.

GC-MS Instrumentation



GC-MS Chromatogram

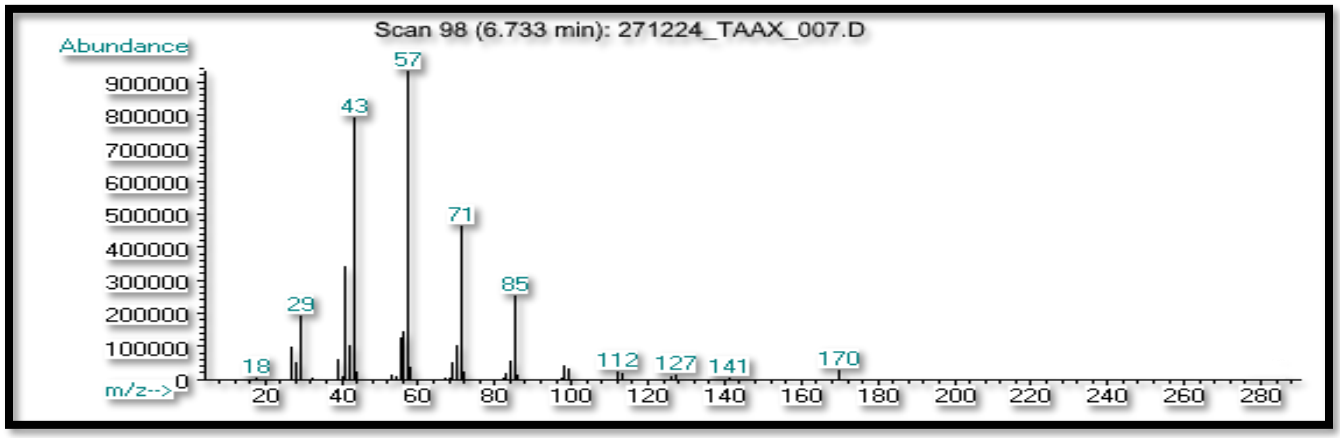
The chromatogram (TIC) below, represents the retention time (x-axis) plotted against the intensity of a signal (abundance). The peaks may represent one or more compounds eluting through the GC Column.



EI Mass Spectrum

As the individual compounds elute from the GC column, they enter the electron ionization. There, they are bombarded with a stream of high-energy electrons (70 eV) causing the molecules to break apart into fragments. These fragments can vary in size with respect to the original molecule. The fragmentation pattern depends on the molecules mass, formula, bonds and geometric shape of the analyte of interest.

Below is the mass spectra of the peak @6.733 minutes.



Compound Confirmation…?

In many cases, care must be taken when confirming the existence/non-existence of a compound. To rely on a single library search may mislead the chromatographer in positively (or negatively) identifying a compound. Below is a typical library search for the above compound. The fact that we added dodecane to the mixture is a reassurance that we have correctly identified the peak @ 6.733 minutes.

Contact the TRACES Manager for full details.