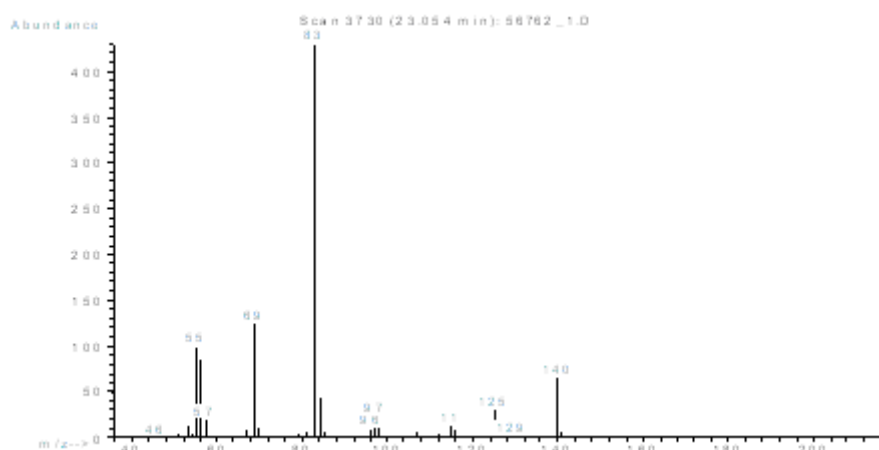# GAS CHROMATOGRAPHY – MASS SPECTROMETRY (GC-MS)

Gas chromatography

Gas chromatography (GC) is a separation technique applicable to compounds which can be volatilised in a gas stream. This is typically done by injection of 0.1-1µL of sample into an injection port heated to 250ºC.   Modern capillary columns allow separation of several hundred compounds in one analytical run as illustrated below.
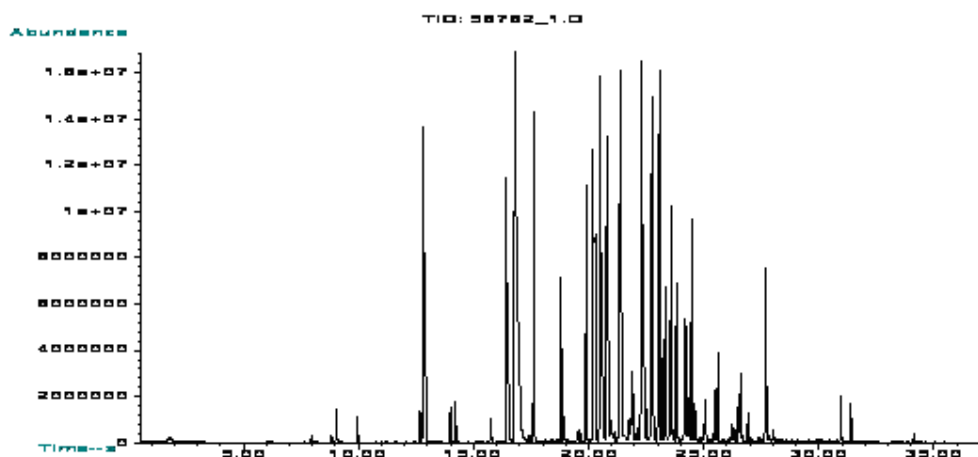
Mass spectrometry

Mass spectrometry (MS) is an identification technique. As compounds elute from the GC column they enter the mass spectrometer where they are bombarded by a stream of electrons which eject electrons from the molecules of compound to produce positive ions. These ions then fragment and rearrange in ways which are very specific to the individual compound. The positive ions and their break-down products are then separated in a magnetic field to produce a "Full Scan Mass Spectrum". The mass spectrum for 3,3,5-trimethylcyclohexanone is illustrated in below.

Gas chromatography-mass spectrometry

Gas chromatography-mass spectrometry(GC-MS) is arguably the most powerful analytical technique available for organic analysis. It is used, for example, at the Olympic Games for drug screening and is now widely used in analysis of environmental samples because of its specificity, selectivity and sensitivity.

A typical GC-MS analysis may take 30-60 minutes with a **Full Scan** (FS) mass spectrum covering 33-450 mass units being collected 1-2 times a second. This generates a tremendous amount of data and requires a powerful computer to process this information.

Different compounds in an analytical run are identified using a number of techniques;

- The retention time of the compound in the GC column is compared to that of standard compounds. Certain qualitative deductions can be made from retention times if standards are not available.

- Library searching. The computer can match the unknown compound's mass spectrum with that in a "library" to produce a list of "best fit" matches. We use the 75000 compound NIST and 350 compound HP Pesticide libraries as well as our own in-house library of spectra generated on our instrument.

Selected Ion Monitoring

Selected Ion Monitoring (SIM) mass spectrometry is a technique used to improve the sensitivity of a mass spectral analysis. Instead of scanning each mass spectrum over a wide range of mass units e.g. 33 to 450, the detector is focussed on only a small number of ions, typically 2 or 3 for each compound. An example of a SIM for the ion of mass 120 of the same sample as in Figure 1 is given in the figure below, showing a much simpler chromatogram.
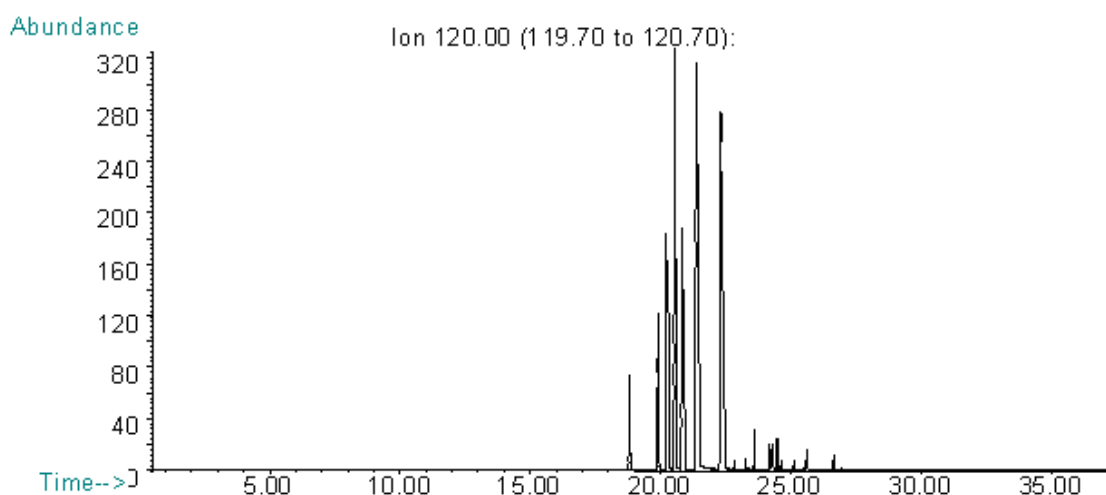
Advantages of SIM

There are two main advantages to SIM MS. Firstly, as the detector can spend more time measuring at the specific ion masses there is an increase in sensitivity, typically 20 to 100 times. The second advantage is that compounds which do not produce the particular SIM ions in their mass spectrum will not be detected and so the chromatogram is much less complex.

To SIM or not to SIM?

We carry out analyses at Hill Labs following USEPA methods, using either Full Scan or SIM as appropriate. The following pros and cons are involved.

Full scan GC-MS

The complete mass spectrum is available for all compounds so that there is a high degree of confidence in the qualitative assignment of peaks to compounds. Compounds which are not available as standards or are not in one of the available libraries can often be identified from their mass spectra. A good technique to use for scanning "unknown" samples and when detection limits are not critical. It is possible to provide lists of "Tentatively Identified Compounds" along with the target compound data. Examples of analyses using FSGCMS are VOC and SVOC.

Selected Ion Monitoring GC-MS

This provides lower detection limits and less matrix interference. Compounds with no ions selected will not be detected. There is a lower degree of confidence in the qualitative identification although this is overcome for USEPA target compounds by using several ions for SIM and comparing their ratios, as well as by matching retention times to that of standards. Examples of analyses carried out using SIM are PAH, PCB and ONP pesticide analyses.

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