GCMS Report Interpretation - Single Page Instructions

Don't be daunted, they get easier after doing a few and you'll learn a lot in the process.
It's only necessary to read this first page – the rest is extra background information.

Peak No.	RT	Area%	Name	CAS#	Score
A peak is usually a single chemical. From: Synthetic sources: Isomers Captives Specialty Bases Additives Natural sources: Isolates Essential Oils Absolutes Resinoids, Gums	**Retention time.** The time the material takes to "elute" from the column. It's pretty consistent in the same conditions, column,gas, temp. etc.	**Quantity** % Area of the peak. An indication of the amount in the compound but materials can have different response factors. So make trials to confirm.	**Raw Material Name** **ID** = Probable identity of the material from spectra matched to a database. **Library** = Databases (DB) used to compare the peaks to. eg. NIST, Adams	**Chemical Abstract Series Number.** Helps to confirm the identity of the material which can have many languages, synonyms or trade names.	**Important** The quality of the match to the DB. % probability it's a correct match. 90 + Very high 80 + High 70 + Probable 50-60 Maybe 30-50 Possible 0-30 Unlikely **BUT** these may support other RMs

- Go through the report peak by peak, one by one, don't rush it.
- Check the quality in the Score column 1 to 99. Maybe highlight all those above 70.
- Make a list of all materials with a score of 70 and above and make a note of the area %.
- If more than 70 then it probably is in the perfume (but not certain).
- Make up your sample using the area % in grams (or drops for quick prototyping).

So 1.00% = 1.00 gram (or 1 drop). Don't worry at this stage that it does not add up to 100.

- 1st sample: When you make your first sample leave out things with a lower score than 70.
- 2nd sample: After you have made your first sample compare it to the original and now consider the other materials that got a lower score. Tip: try them on smelling strips first.
- 3rd sample: Try replacing some materials with the essential oils you think they could be.

GCMS REPORT TIPS:

- Materials with a low score are likely there if also linked to others with a higher score. If a
- name ends in …..**ene** (e.g. pinene, limonene, cymene, terpinolene, myrcene, patchoulene etc.) it is probably a terpene or sesquiterpene from an essential oil. There is no one easy way to tell which oil. You have to look at other peaks like Citral in Lemon, Decanal in Orange, Linalool and Linalyl Acetate in Bergamot or Lavender.
- Some materials have **isomers** which means more than 1 peak. (DPG has 4, Triplal and Rose Oxide 2, Citral 2 (Geranial and Neral), Iso E Super, Vertofix, Galaxolide, Methyl Ionone, Indisan, Hercolyn D, Patchouli oil etc. have many, many peaks. Add the quantities together to get the total amount of each material or oil.
- If you see "**diethyl acetal**" or "**DEA**" these are probably from reactions with alcohol in the product. Just add the quantity of them to the main product. Lyral + Lyral Diethyl Acetal, Lilial + Lilial Diethyl Acetal, Hydroxycitronellal + Hydroxycitronellal DEA etc.
- If you don't know the material check the name or CAS number by searching in Google "***Goodscents Name***" or "***Goodscents CAS 115-70-3***"
- Matches from the Wiley or NIST databases are less reliable as they may not even be fragrance materials. Just leave these out initially if the quality is low.

OPTIONAL BACKGROUND INFORMATION

GC - Gas Chromatography (or GLC - Gas Liquid Chromatography)

Gas Chromatography Is a method of separating a compound usually for the purpose of identifying the component chemicals. The separation occurs according to each component chemicals affinity for a moving gas phase or for a stationary phase (e.g.. wax or silicon packing material). A very small amount of the compound (e.g. 1 microlitre/ 0.001 cc) is injected into the flow of a hot gas at the beginning of long (the one we use is 60 metres long) very narrow (capillary) tube (the column). According to the affinity of each chemical ingredient for the gas or for the stationary phase each chemical moves at a different rate along the column. This effectively means that each chemical comes out (elutes) at a different time. As the chemical emerges from the tube it is detected. In very accurately controlled conditions every chemical has a specific 'retention time' (RT) which helps to identify it.

Some terms you will come across with reference to GC:

Polar / Non Polar Columns - the packing in the column is chosen for the best separation (resolution) of the sample. A polar stationary phase such as Carbowax will slow a chemicals movement along the column if that chemical has a charge. A non-polar column such as Silicon (DB5) is far more inert and chemicals tend to move along according to their molecular size - smaller molecules coming out first followed by larger ones.

The Gas Chromatogram
The print out seen in the Workflow Report where the peaks of eluting components from the GC column are plotted peak area vs. retention time the one used here is from an Agilent GC using a Mass Spectrometer detector with their Mass Hunter Software.

KI - Kovat's Index
A standard list of RRT - relative retention times, named after the originator from the '60's useful benchmarks for GC owners to help set up their own equipment.

FID - Flame Ionisation Detector
As the material is eluted (emerges) from the column in enters a flame supplied from air and hydrogen cylinders. The ionisation (charged particles) of the flame is measured by two electrodes connected to an amplifier and recording device (chart recorder or "integrator"). As the chemical mixed in the column gas enters the flame the chemical alters the ionisation and the change is detected. This then triggers the needle on the chart recorder to move and the higher the concentration of chemical the higher the needle moves. The FID detector only allows us to register the presence of something in the gas but tells us nothing about its identity.

Olfactometry
One useful GC technique, unique to flavour and perfumery, is the splitting of the column for GCO - GC Olfactometry. Here the column in the GC is split into two near the end so that part of the sample goes to the FID or MS and the other leads out of the GC to a small funnel. The funnel should be fitted with a heat-sink to aid cooling. The perfumer then simply sniffs the eluant from the GC column and attempts to identify each material by its smell. He knows when the main peaks are coming as he watches the other detector responding to its presence. Sometimes GCO will identify materials at levels so low that they do not even produce a peak on the chromatogram.

MS - Mass Spectrometry
The eluant from the column of the GC is fed into a Mass Spectrometer. There under near vacuum the molecules in the gas are bombarded with a stream of electrons or pass over a filament that breaks the molecules up into characteristic fragments. These fragments have a charge due to losing electrons then pass into a strong but finely controlled quadrapole (4 pole) electro-magnetic field where each fragment is pulled and deflected, according to its mass vs. charge, so it hits a detection plate. These hits produce a pattern, the mass spectrum. This spectrum is like a fingerprint which can then compared on a computer database to predict the identity of the molecule.

Some other definitions of GC (Encyclopedia Britancia etc.)

- The Gas Liquid Chromatograph is a system in which a liquid with a high boiling point is impregnated on an inert solid support packed into a thin metal column, and helium gas is allowed to flow through it. The solution to be analyzed is injected into the column, immediately volatized, and swept by the helium gas through the column; at this point the mixture is resolved into its components.
- Gas Chromatography - in analytical chemistry, technique for separating chemical substances in which the sample is carried by a moving gas stream through a tube packed with a finely divided solid that may be coated with a film of a liquid. Because of its simplicity, sensitivity, and effectiveness in separating components of mixtures, gas chromatography is one of the most important tools in chemistry. It is widely used for quantitative and qualitative analysis of mixtures, for the purification of compounds, and for the determination of such thermo-chemical constants as heats of solution and vaporization, vapour pressure, and activity coefficients. Gas chromatography is also used to monitor industrial processes automatically: gas streams are analyzed periodically, and manual or automatic responses are made to counteract undesirable variations. Many routine analyses are performed rapidly in medical and other fields. For example, by the use of only 0.1 cubic centimetre (0.003 ounce) of blood, it is possible to determine the percentages of dissolved oxygen, nitrogen, carbon dioxide, and carbon monoxide. Gas chromatography is also useful in the analysis of air pollutants, alcohol in blood, essential oils, and food products.
- The method consists of, first, introducing the test mixture or sample into a stream of an inert gas, commonly helium or argon, that acts as carrier. Liquid samples are vaporized before injection into the carrier stream. The gas stream is passed through the packed column, through which the components of the sample move at velocities that are influenced by the degree of interaction of each constituent with the stationary non-volatile phase. The substances having the greater interaction with the stationary phase are retarded to a greater extent and consequently separate from those with smaller interaction. As each component leaves the column with the carrier, it passes through a detector and then either goes to a fraction collector or is discarded.

MS - Mass Spectrometry

The eluant from the column of the GC is fed into a Mass Spectrometer. There under very low pressure the aroma molecules in the gas are bombarded with a stream of electrons and break up into characteristic fragments. These fragments then pass into a strong magnetic field where each fragment is deflected differently, according to its mass, to produce a pattern, the mass spectrum. This spectrum is like a fingerprint which can then compared on a computer database to predict the identity of the molecule.

MS -Mass Spectrometry Background Info

Also called MASS SPECTROSCOPY, analytic technique by which chemical substances are identified by the sorting of gaseous ions in electric and magnetic fields according to their mass-to- charge ratios. The instruments used in such studies are called mass spectrometers and mass spectrographs, and they operate on the principle that moving ions may be deflected by electric and magnetic fields. The two instruments differ only in the way in which the sorted charged particles are detected. In the mass spectrometer they are detected electrically, in the mass spectrograph by photographic or other nonelectrical means; the term mass spectroscope is used to include both kinds of devices. Since electrical detectors are now most commonly used, the field is typically referred to as mass spectrometry.

Mass spectroscopes consist of five basic parts: a high vacuum system; a sample handling system, through which the sample to be investigated can be introduced; an ion source, in which a beam of charged particles characteristic of the sample can be produced; an analyzer, in which the beam can be separated into its components; and a detector or receiver by means of which the separated ion beams can be observed or collected.

Many investigations have been conducted with the help of mass spectrometry. These include the identification of the isotopes of the chemical elements and determination of their precise masses and relative abundances, the dating of geologic samples, the analysis of inorganic and organic chemicals especially for small amounts of impurities, structural formula determination of complex organic substances, the strengths of chemical bonds and energies necessary to produce particular ions, the identification of products of ion decomposition, and the analysis of unknown materials, such as lunar samples, for their chemical and isotopic constituents. Mass spectroscopes also are employed to separate isotopes and to measure the abundance of concentrated isotopes when used as tracers in chemistry, biology, and medicine.

Chromatography-mass spectrometry methods

The mass spectrometer is an analytical instrument that bombards molecules with a stream of electrons in a chamber at extremely low pressure to produce a stream of charged fragments that differ in mass (see the article mass spectrometry). The population of the fragments and the ratio of mass to charge is characteristic of the target molecule. Each fragment is deflected differently in a magnetic field to produce a pattern, the mass spectrum, which can be used to identify the target. The system is a very specific identifying detector when coupled with chromatography. The spectrum can
be stored in a computer and compared with entries in a mass spectrum library.

For some time the problem with gaseous effluents had been to match the column effluent at one atmosphere pressure to the high-vacuum inlet of the mass spectrometer, while the problem with liquid chromatography had been the large amount of mobile phase entering the ionization chamber of the spectrometer. These incompatibility problems have finally been overcome, and the mass spectrometer is now used in both gas and liquid chromatography. The technology of mass spectrometry is as great, if not greater, than that of chromatography.

If mass spectral data are lacking, solutes in a sample are identified by comparing their behaviour with that of known compounds. In gas chromatography this is best done by determining the retention index of the unknown solute and comparing it with the tabulated data for known compounds on the stationary phase used. Methods exist for estimating the effect of temperature and temperature programming on the retention index.

The area enclosed by a peak, suitably adjusted for the detector response factor for that solute, is proportional to the amount of solute producing the peak. The area is frequently approximated from the peak width and height. Modern electronic integrators will, when properly instructed, ignore electronic noise, compensate for baseline drift, start integration when a peak appears, integrate, and stop the process when the peak exits the detector. Integration, a process of summation, is accomplished by opening and closing a narrow electronic window, registering the signal intensity,

repeating the process, and then summing the stored signals to produce a number proportional to the area. The integrator will also sense the peak maximum. The chromatogram is a printed tape with the retention times and peak areas. Programs exist that will incorporate the response factor and calculate the relative peak areas, which give the percentage composition of the sample. Stored mass spectral data may be manipulated to produce the same data. Peak heights are used as quantitative
measures for narrow peaks for which the area is difficult to determine accurately.