**Gas chromatography in food analysis: an introduction**

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**Abstract**
Introduces gas chromatography (GC), a key analytical technique in the food industry. It enables complex organic substances to be separated and identified quickly and cheaply. Substances to be analysed by GC must be volatile, i.e. readily pass into the gas phase. The substance to be analysed is vaporized and moved through a long column by an inert carrier gas. The column is filled with a packing material covered with an involatile liquid. The molecules of each substance in a mixture will become distributed between the gas and the liquid. The more volatile a substance the longer it will be moving with the carrier gas, and the quicker it will emerge from the column. Some substances must be extracted from the food analysis by GC, e.g. fatty acids from triglycerides. Others, such as alcohols, can be injected directly into the column.

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**Introduction**
Gas chromatography (GC) is an analytical technique widely used in the food and drink industry. It is capable of rapidly separating and detecting the volatile organic components of a food or drink. A volatile substance is one that readily passes into the gas phase; normally these are liquids or solids with low boiling and melting points. A good example would be ethanol which is present in alcoholic drinks.

GC is particularly valuable when complex mixtures need to be analysed. During the quality control process in food production a processor may wish to check if toxic substances such as pesticide residues are present. Chemical analysis using traditional techniques would be slow and laborious, but by using GC it is possible to analyse to detect many such substances in a few minutes. Advanced GC systems can easily separate and detect 100 different compounds in about one to two hours. The analysis of the flavour components of herbs was a very time-consuming process before the advent of GC, but when GC was applied to the analysis of the flavour components from coriander leaves, it was possible to separate and detect the 30 major aromatic components in about 60 minutes.

**Principles of GC**
The basis of the separation is a retardation of the individual components as they are moved through a long column by a carrier gas, usually helium or nitrogen. The column consists of a steel or glass tube filled with an inert packing material such as glass or ceramic beads (see Figure 1). In gas-liquid chromatography (GLC), these are coated with an involatile liquid, so that the surface area of the liquid in contact with the gas is large. For some applications, the packing may be a solid without any liquid coating; it is then called gas-solid chromatography (GSC), but this is less widely used than GLC.

The sample is injected into the carrier gas stream. As it moves through the column with the carrier gas, the molecules of each substance present in the sample will distribute between the gas and the liquid. Individual molecules will constantly move between the gas and the liquid in a dynamic equilibrium. While a molecule is in the gas phase it will pass along the column, while it remains...
dissolved in the liquid it will be stationary. The more volatile a substance, the greater proportion of time its molecules will be moving in the carrier gas, and so the sooner it will emerge from the column. In this way each substance will become separated within the column and emerge separated by time at the end.

The time taken from injection to emergence is known as the retention time ($R_t$) (see Figure 2), and is characteristic for each substance under any given set of conditions. It depends on the volatility of the substance, as well as the temperature of the column and its length and diameter.

Many substances have an inconveniently long retention time at room temperature, and this is overcome by heating the column in an oven. Having separated the components in the column so that they emerge individually, some method of detecting and measuring them is needed. Two types of detector are commonly used: thermal conductivity and flame ionization.

Thermal conductivity detectors (TCDs) rely on changes in the thermal conductivity of the gas leaving the column. The pure helium carrier gas passes over a hot tungsten-rhenium filament, causing it to cool, since helium has a very high thermal conductivity. When a chemical substance emerges with the carrier gas, cooling will be less, and the temperature of the filament will rise. As with most metals, its electrical resistance increases with temperature and this can be measured and recorded.

Flame ionization detection (FID) is more frequently encountered in food applications, since many compounds under investigation are organic (containing carbon), and FID is around a thousand times more sensitive than thermal conductivity detection for organics. The gas emerging from the column is burned with a hydrogen and air mixture. This forms ions, which conduct an electric current which can be amplified and recorded on a chart recorder. Although the number of ions formed in this way is small, perhaps only 0.0001 per cent of the total carbon atoms present in the sample, the proportion produced is always constant. This means that the total signal recorded on the chart recorder is proportional to the amount of the chemical substance present.

Applications in food analysis

Many of the substances commonly analysed by GC need some kind of extraction from the food sample before analysis. Direct injection would cause non-volatile solids to be deposited on the column, and would quickly contaminate it. For example, determining the fatty acid content of triglycerides (fats and oils) is an important use of GC, but they are not volatile enough to pass through the GC column. They must first be converted into a more volatile derivative which still retains the identity of the original fatty acid. Since a triglyceride is an ester of fatty acids and glycerol[1], one method of achieving this is to split the triglyceride into fatty acids and glycerol. The fatty acids are then converted into methyl esters of fatty acids[2], which are easily separated from the glycerol by dissolving them in a solvent. The methyl esters and the solvent can then be injected directly into the chromatograph, and are easily separated and detected.
With increasing public awareness of health issues relating to saturated fats and trans fatty acids in the diet, manufacturers are using GC to determine the percentage of saturated fatty acids and trans fatty acids in margarines, oils and spreads. GC also has an important role in determining the percentages of essential fatty acids in triglycerides consumed by the population, and hence whether the average diet contains sufficient essential fatty acids for good health. A typical chromatogram for a vegetable oil is shown in Figure 2.

An interesting application of GC relies on the different ratio of fatty acids present in the body fats of each animal. This makes it possible to tell exactly what meats are contained in for example, a burger; is it beef, or has some other animal been used? Kangaroo meat has been discovered in burgers in this way in the past!

For some applications, direct injection into the GC column is possible, for instance in analysis of alcoholic drinks. Wine can be sampled by direct injection of air from the headspace (the air above the wine in a sealed container) into the chromatograph. Analysis using headspace volatiles measures only those components volatile at room temperature, and is therefore most useful for studying the bouquet development. It has great potential as a way of deriving a synthetic aroma similar to the bouquet of wine.

Australian researchers have used GC to develop a method of identifying counterfeit brandies, which have been made by the fermentation of something other than grapes. This is often a cheaper material, such as other fruits or cereal starch. They found that the ratios of some of the alcohols present in brandy were more important than the individual quantities of any substance in it. The ratio of butyl alcohol (C₃H₇CH₂OH) to propyl alcohol (CH₃(CH₂)₂CH₂OH) was a key factor, and their analysis of brandies known to be of pure grape origin suggested a maximum of 5:1 for this ratio. Cheap brandies suspected as being counterfeit had ratios of up to 26:1, while the more expensive ones had ratios around 3:1.

**Conclusions**

The discovery of gas chromatography revolutionized the food industry, and is a standard item in most laboratories involved in food testing. It has an important role in quality control, enabling buyers and trading standards authorities to ensure the product is as stated on the label. Health checks for toxins, for example, pesticide residues in fruit and vegetables, are another vital area. GC is used to check that levels of pesticide are below limits currently considered safe.

Increasingly, GC has a role in the development of synthetic flavours and aromas. If the exact chemical composition of a flavour can be quantified, it may be possible to manufacture a synthetic copy, identical to the naturally occurring product, but much cheaper to produce to a consistent quality. GC can, however, only be used to compare an unknown component with a known standard. To identify an unknown substance, a range of more complex analytical techniques may be applied to the separated substances from the gas chromatograph. These include GC-mass spectrometry, and GC-infrared spectroscopy.

**References**