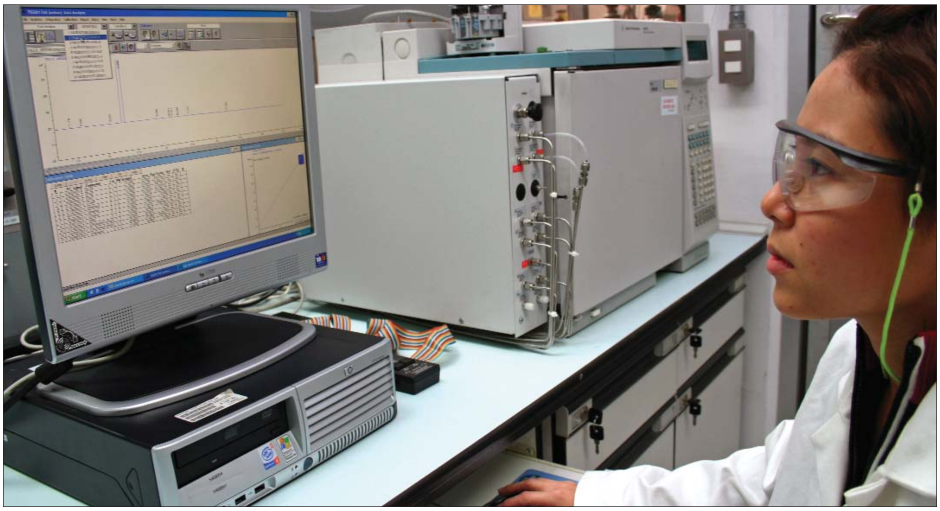
Gas Chromatography—Mass Spectrometry

The Quantity and Quality Detector

By Stephen Harrison, Linde Gases



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Gas chromatography-mass spectrometry (GC-MS) is one of the most important tools in analytical chemistry. Where other analytical techniques determine quantitative issues arising from analysis of a sample (how much of a chemical is present), GC-MS is able to identify qualitatively the nature of chemicals in the sample, identifying which molecules are present. According to the GC principle, molecules in a sample separate in the chromatography column due to differences in their chemical properties. Mass spectrometry breaks components into ionised species and separates these based on their mass to charge ratio. Gas chromatography is the first separation step and mass spectrometry is the subsequent step that performs qualitative detection.

"Amid heightened concerns about food safety in many parts of the world *(see related story "The Food and Beverage Market: Food Safety Initiatives Usher in New Regulations" on p. 58)*, GC-MS has become an important analytical technique," says Stephen Harrison, Linde Gases' Head of Specialty Gases and Specialty Equipment. "It is one of few techniques to determine exactly what is in a food sample. Characterized by its quick screening abilities, GC-MS has been widely heralded as the 'gold standard' for forensic substance identification."

Like GC-MS, liquid chromatography-mass spectrometry (LC-MS) is a qualitative analytical chemistry technique combining the physical separation capabilities of high performance liquid chromatography with the qualitative analysis capabilities of mass spectrometry. GC-MS is used to screen a sample using a gaseous phase component separation process in the gas chromatography column, while LC-MS is able to detect and identify chemicals using a liquid phase component separation process in the liquid chromatography column. The medium in which the sample exists and is most effectively separated in the chromatography column—gaseous or liquid—determines which technique is more appropriate. GC-MS is preferred when quick screening is required because the column separation is generally faster in the gaseous phase.

GC-MS is commonly used to screen for contaminants in food and drugs, and we were reminded of its relevance in Europe recently after elevated levels of dioxins showed up in small amounts of German eggs. This sophisticated scientific technique, which is routinely used by the Food and Drug Administration in the US to detect dioxin, can be successfully employed to trace the source to a single manufacturer. European Union rules allow no more than a one part per trillion of dioxin in food for human consumption. Dioxin and chemically-related compounds are a group of environmental contaminants found worldwide.

In reference to the dioxin problem in Europe, Harrison says, "Applying a qualitative technique in a detective-like fashion in this crisis situation was critical. Samples could be checked right back through the food chain until analysts reached a point where the chemical did not show up in the sample—indicating that this is where it entered."

Similar scares have occurred in the beverage industry. One of the most notable was benzene found in soft drinks in the US in the 1990s. Benzene levels are regulated in drinking water nationally and internationally, and in bottled water in the United States, but only informally in soft drinks. Benzene, which is carcinogenic, results from decarboxylation of the preservative benzoic acid in the presence of ascorbic acid (vitamin C), especially under heat and light. Alcohol can be another unregulated beverage. In countries where spirits are made in unlicensed stills, beverages may contain toxic alcohols such as methanol, which can cause blindness and death. GC-MS or LC-MS in the hands of food safety officials would protect consumers from potentially unhealthy or lethal effects of substances like benzene and methanol in beverages.

Pharmaceuticals

In the pharmaceutical industry, drug content safety has high priority. GC-MS is used to avoid contamination, check for appropriate active ingredients, and maintain customers' trust in pharmaceutical authenticity. "Doping in top level sporting events is a pharmaceutical scenario where GC-MS is a powerful forensic technique," says Harrison. To prove athletes are free of performance enhancing drugs, blood and urine samples are run through a qualitative technique to see if any new or unexpected molecules are present. These unbiased results can be presented as critical evidence in a court of law, because they prove exactly what substances were present. To back up this evidence, a quantitative analysis will reveal how much of the substance is present. The combination of qualitative and quantitative analysis is key.

Harrison adds, "GC-MS also can play a significant role in industrial production, analyzing the composition of process streams. In industry, things can change suddenly and put an entire process at risk—an unknown molecule may infiltrate the process stream for the first time. GC-MS can identify the exact chemical causing the problem. It is perfect for troubleshooting in these cases. When it comes to research, investigation, innovation, discovery, and trouble-shooting industrial processes, GC-MS can be a key approach, particularly at the beginning of a new process or during process changes and enhancements."

Origins

The use of a mass spectrometer as the detector in gas chromatography was developed during the 1950s by Roland Gohlke and Fred McLafferty in the United States. The first devices were bulky, fragile, and limited to laboratory settings. The development of affordable and miniaturized computers and other instrument parts helped make this instrument easier to use and allowed great improvements in the amount of time it takes to analyze a sample. For example, in 1996, top-of-the-line, high-speed GC-MS units completed analysis of fire accelerants in less than 90 seconds, whereas a first-generation GC-MS would have required at least 16 minutes. Improvements like these have led to the widespread adoption of GC-MS units in a number of fields in the past two decades.

"The first gas chromatography mass spectrometer I saw filled a large room," says Harrison. "Today, you can put a GC-MS into a small suitcase. As a result of rapid evolution in portability, you can now analyze samples at source, which always makes for an optimum result.

"As a supplier of gases for GC-MS, Linde has developed technology to keep up with changing modes of gas supply. When GC-MS units were huge machines we supplied essential operating gases in standard 40 or 50 liter cylinders or by CRYOSPEED® liquid gas supply. When GCs became 'micro-sized,' we came up with the HiQ® MICROCAN, a beer bottle-sized cylinder containing enough gas to keep a GC-MS running for several months."

High Purity

GC-MS requires high quality specialty gases for instrument operation and for calibration. Specialty gases are used as carrier gases (helium is most common), collision gases (nitrogen or helium), and can also include reagent gases. The carrier gas plays an important role by transporting the sample through the chromatography column into the mass spectrometer. The carrier gas must be inert or at least must not react with the stationary phase in the column. The choice depends on the sample, column, application, and safety requirements.

The choice of carrier gas is also dependent on requirements in terms of separation efficiency and speed. Hydrogen has the lowest viscosity of all gases, so provides the highest mobile phase velocity and therefore the shortest analysis time. Helium, on the other hand, gives the best overall performance and peak resolutions for many applications, making it an optimum choice of carrier gas in those cases.

The purity of the carrier gas is critical for performance, maintenance, and lifetime of the column. Impurities, especially hydrocarbons, cause base line noise and reduced sensitivity and might increase detection limits. Traces of water and oxygen may also decompose the stationary phase, which leads to premature destruction of the column. Linde's gases range in purity up to 7.0, which is the highest commercially available grade, being 99.99999 percent pure with 0.1 parts per million of total impurities. The gas company also provides certificates of analysis on its own gases to reassure customers of these exceptional quality attributes.

"While the reliability of analysis is only as good as the quality of gas being used, distribution systems and equipment for high purity gases and specialty gas mixtures must also be able to meet increasing demands for high standards of performance and new analyzing methods," comments Harrison. "Impurities occurring in concentrations as low as parts per billion can have serious consequences, particularly if the analyst is not sure which molecules are present in the sample.

"Demands made on regulators and valves in these environments are extremely high. Components must be capable of dealing with high and low pressures, large and small flows. They must be suitable for high purity inert gases as well as reactive, flammable, corrosive, or toxic gases."

Linde's hi-tech range of regulators, REDLINE®, is modularly designed to slot into central gas supply systems containing gas panels, and points-of-use and cylinder regulators generally suitable for purities up to 6.0 (99.9999 percent). Linde also has HiQ® laboratory gas generators providing high purity hydrogen and nitrogen for carrier gas suitable for GC-MS.

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