Process Mass Spectrometry

A Tool for High Performance Gas Analysis

By Graham Lewis

Mass spectrometers (MS) have been providing fast, precise online gas analysis for industrial applications for more than 30 years and continue to provide valuable data for process control and environmental and safety monitoring. But one trend in particular — accessibility — has had a substantial impact on the recent growth of these instruments.

Traditionally regarded as a powerful but delicate laboratory tool relegated to the realm of highly-trained lab experts, gas analysis mass spectrometers have become more available to experts and non-experts alike as the technology behind these instruments has become demystified. Process mass spectrometry — specifically magnetic sector-based technology — has brought added practicality and efficiency to gas analysis applications.

Why Use MS for Gas Analysis?

Mass spectrometry has long been one of the preferred methods for multi-component gas analysis because of its speed and range; gas analysis mass spectrometers can provide a quantitative analysis in seconds and are more flexible in terms of analytical capability than most other analyzers. These tools have been used to monitor process streams in the petrochemical and chemical, pharmaceutical, and biotechnology, and iron and steel manufacturing industries, among others, for years due to their ability to analyze an extremely wide range of concentrations of inorganic and organic gases.

Besides the unique speed and range of mass spectrometry in industrial gas analysis applications, there is another key difference between mass spectrometers and other gas analyzers. Mass spectrometers are much more flexible because their analytical capabilities are mainly defined in software, compared to many other gas analyzers whose analytical capabilities are defined in hardware. This distinction is what allows an MS to analyze a wide range of sample streams with widely different compositions. For example, in the chemical and iron-making industries, the MS can analyze natural gas with methane concentrations over 90 percent as a feedstock, then later in the process measure streams containing methane at less than 0.5 percent concentration.

How It Works

All mass spectrometers share the same five basic components, whether they are analyzing solids, liquids, or gases.

Sample selection: The two main types of sample selectors for gas analysis are continuous inlet and batch inlet. A continuous inlet is used when a relatively large amount of sample is available to flow to the MS. Sample selection is under software control, with rapid multi-stream samplers, rotary valves or banks of solenoid valves used to switch between gas samples. Because of the speed of MS, it is not uncommon to have up to 60 sample streams being analyzed by one MS. The batch inlet is used when a limited amount of sample is available, usually at low pressure. The sample is expanded into a fixed volume to reduce its pressure, and then into the MS.

Pressure reduction: In most applications it is preferable to ensure that the original sample composition at the original sample pressure (usually atmospheric or slightly above atmosphere) is maintained at the vacuum pressure in the MS: the pressure reduction system should not discriminate between lighter and heavier atoms and molecules. The most common inlet is a combined capillary and molecular leak, and an alternative is the membrane inlet.

Ionization: The most common technology for ionizing gas samples is electron impact (EI). In EI, streams of high energy electrons are emitted from a hot filament and focused to collide with the sample gas; these collisions eject electrons and produce positively charged ions, which are directed into the mass separator by electrodes. While EI is relatively simple and extremely well-proven, detection of trace components can be dif-

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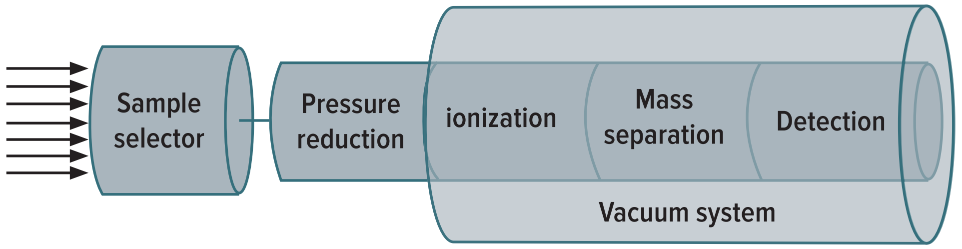
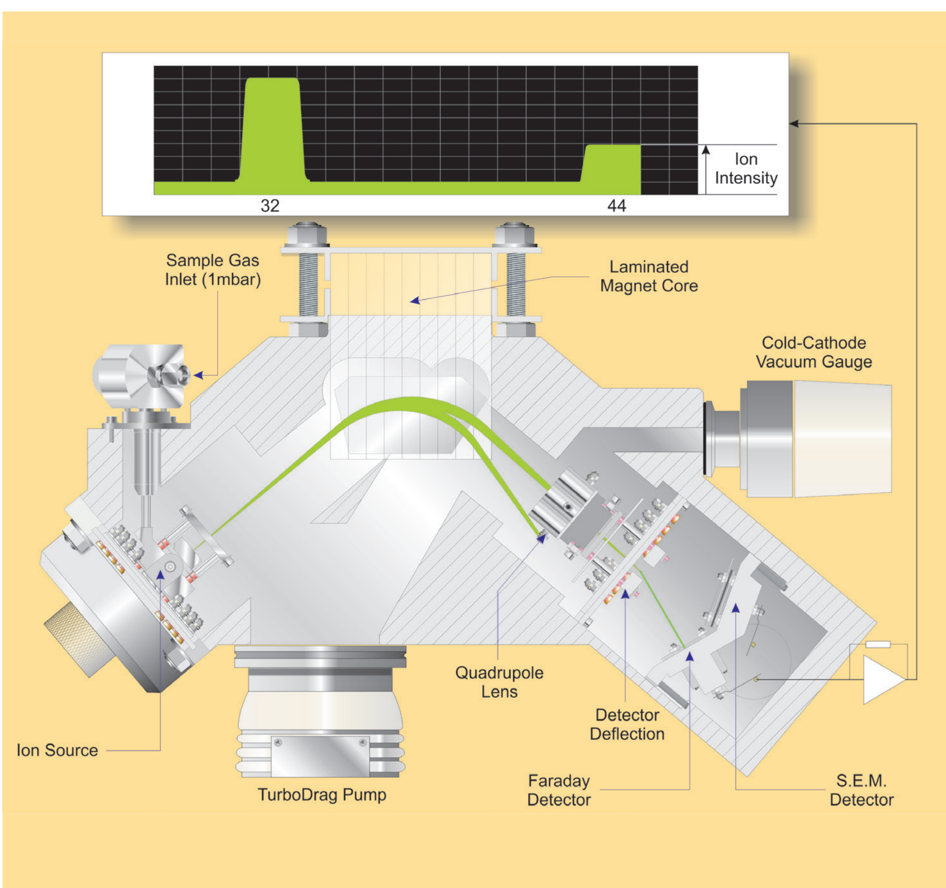


ficult, so other techniques — including atmospheric pressure ionization, chemical ionization and proton affinity ionization — are sometimes used for specialist applications.

Mass Separation: There are two main types of MS analyzer — magnetic sector and quadrupole — used for process gas analysis. In a magnetic sector MS, ions are accelerated through a flight tube, where they are separated by their mass to charge ratios in a magnetic field of variable strength. In a quadrupole MS, ions are separated by their trajectories in oscillating electric fields that are applied to the rods.

The magnetic sector MS provides greater stability and precision (between two and 10 times better) than the quadrupole MS. Unlike the flat-topped peak generated by the magnetic sector, the quadrupole produces a Gaussian 'round top peak.' So it is 'fault sensitive' — any drift in the mass scale will produce an error in the peak height measurement. This has to be corrected by more frequent calibration.

The magnetic sector MS is also much more resistant to contamination than the quadrupole. The ions are accelerated into the magnetic sector MS with high energy so will not be deflected by small field effects caused by contamination. The ions entering the quadrupole analyzer are accelerated with low energy and are therefore much more easily deflected by contamination build up. In the early stages, this can be corrected by recalibration, but over time the contamination will need to be removed by cleaning. The intervals between cleaning can be relatively short if the sample streams contain high levels of hydrocarbons.



Detection: Most gas analysis applications are carried out using a Faraday cup detector, which has a very wide dynamic range. A secondary electron multiplier may be used when greater sensitivity is required. A modern gas analysis MS may employ both detectors for high concentration and low concentration ion beams.

Conclusion

Smaller. Faster. More precise and intuitive. Technology has advanced dramatically and rapidly over the last two decades, including in the world of mass spectrometry. Scientists and researchers can evaluate more processes faster, which accelerates development and innovation. Quality by Design (QbD) principles ensure rigorous testing, minimizing process disruption once instruments are online.

Today's magnetic sector mass spectrometers, such as the Thermo Scientific Prima PRO process analyzer and Prima BT benchtop analyzer, allow users to ensure high levels of precision without sacrificing in other areas, including speed. The ease of use, time savings, and cost savings have afforded more industries and more personnel the chance to maximize the usage of gas analysis through a process MS. SGR

About the Author

Graham Lewis is a technical consultant with Thermo Fisher Scientific. He has a degree in Chemistry from Liverpool University, UK, and has worked to promote the use of process mass spectrometers in various industries, including pharmaceuticals, petrochemicals and iron and steel for nearly 30 years. He is based at Thermo Fisher Scientific's process mass spectrometer factory in the UK.