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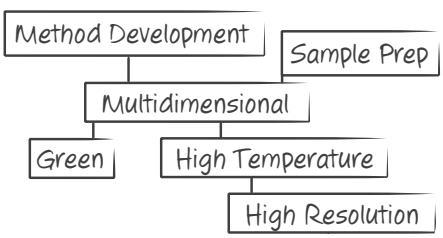

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Please visit our facebook page: *www.facebook.com/AnalyticalChemistryTechniques*

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INSIGHTS
LAB TECHNOLOGY BUYER'S REPORT

INSIGHTS ON GAS CHROMATOGRAPHY SYSTEMS

STILL THE WORKHORSE FOR ORGANIC CHEMICAL ANALYSIS

Despite steadily losing ground to high-performance liquid chromatography (HPLC) over the years, particularly for polar compounds, gas chromatography (GC) remains one of the more rapid and efficient chromatographic methods. Where LC has emerged as the platform of choice for the life sciences, GC remains the standard for "organic" chemical analysis of relatively low molecular weight compounds of medium to low polarity.

Despite being a mature technology, gas chromatography systems were experiencing modest growth in the global market before the current recession. A report by Global Industry Analysts (San Jose, CA), *Gas Chromatography Systems – a Global Strategic Business Report*, suggests that companies deferred plans to purchase or upgrade GC systems during the downturn but will resume buying as the economy improves.

> "GC systems are estimated to reach $1.2 billion worldwide by 2015."

▲ *GC-TOF System | AccuTOF GCv|4G | JEOL | www.jeolusa.com*

Interestingly, Europe represents the largest market for GC systems, about 30 percent of the global market, followed by the United States and Japan. According to the report, significant growth is expected in developing countries in the Asia-Pacific region. Still, this "fastest-growing market" will increase at only about 2.2 percent per year. All told, sales of GC systems are estimated to reach $1.2 billion worldwide by 2015.

The report provides few surprises as far as industry segments most involved in GC: chemicals, pharmaceuticals, and petrochemicals (the fastest-growing industry segment). GC users seek the same types of enhancements and workflow improvements as do other instrument specialists, according to the report: improved resolution, more-rapid analysis, higher sensitivity, ease of use, and enhanced, reproducible measurements.

The issues affecting GC markets and end users on the operational side—throughput and productivity—overlap with other instrument categories. For instrumentation, the leading concerns are stationary phase (columns and chemistries), mobile phase (carrier gas), detector, and maintenance.

▲ *GC Systems | SCION™ Series Bruker | www.bruker.com*

EFFICIENCY AND PRODUCTIVITY

"GC users continue to experience the drive toward improved efficiency and productivity," observes Eric Denoyer, Ph.D., marketing director for GC and workflow automation at Agilent Technologies (Santa Clara, CA). "Managers are being asked to do more with less time, funding, staff, and skill."

"Fast" or "rapid" GC is one way to realize these goals. To shorten run times, analysts are adopting microbore columns, which in turn is driving improvements in low-volume, high-precision liquid autoinjection. Low-thermal-mass devices ramp thermal profiles faster and cool columns more rapidly as well. Denoyer refers to temperature cycling as a "major time hog, especially as GC run times shorten." Many users are

"Autosamplers are the most used and most significant automation upgrade for GC."

also considering switching to hydrogen from denser, more expensive carrier gases to shorten elution times. These improvements have led to shortening of analysis times, for some applications, by a factor of three or more.

Industry's obsession with lean staffing has greatly shrunk the pool of GC technical expertise, with remaining holdouts residing mostly at corporate centers of excellence. This has given rise to analyzer "solutions" (versus standalone instruments) being bundled with methods and spectral libraries, which combined reduce start-up, method development, and validation efforts. "Smarter instruments that are more self-aware of their configuration and operating status can help even less-skilled users plan maintenance downtime and avoid costly unplanned shutdowns," according to Denoyer.

SAMPLE PREP AND AUTOMATION

To speed sample prep and reduce maintenance due to contaminating matrix and nontarget components, analysts are turning to solid-phase extraction or microextraction to reduce inlet and liner contamination. Also, backflush is becoming more common for reducing column and MS contamination and reducing sample cycle time.

In instances where labs perform the same separations under the same methods at high throughput, sample prep automation can make a lot of sense. In those cases, labs look for more of a complete package than separate instruments, says Dan Carrier, an applications chemist at Anatune (Cambridge, UK), which specializes in GC sample preparation hardware and systems. Anatune packages chromatographs and sample prep hardware from vendors into a "solution" that includes the GC, the detector, the automation component, prepackaged methods, and application advice.

"Most of these solutions involve some aspect of sample prep that includes either enriching sample in the target analyte, removing the matrix, or both," Carrier says.

Paradoxically, economic downturns can be a boon for costly automation equipment, as companies can compensate for workers they let go by acquiring automation. "It might add between 25 to 50 percent to the cost of a basic GC," Carrier adds, "but in the long run, automation can actually save money."

Autosamplers are the most used and most significant automation upgrade for GC, but also the component most prone to failure. Autosamplers have more moving parts than all the remaining components combined and are mechanically the most complex equipment within a system.

Automated sample prep, typically involving a liquid-handling robot, is another feature that high-throughput labs should consider. GC samples come from remarkably varied environments. Ensuring that samples undergo reproducible cleanup is perhaps the most significant quality operation. An almost limitless list of interfering species exists in most raw sample streams, which often requires a degree of human intervention—even with automated sample prep.

What is the tipping point for automating or not automating? Every lab manager must calculate return on investment based on time saved compared with manual operation, as well as the value of consistency and added throughput. Cost-benefit analyses are more straightforward for autoinjectors than they are for sample prep because of the diversity and complexity of GC samples themselves.

"But no matter the skill or staffing level, analyzing active or thermally labile compounds at trace levels remains a challenge in many applications," Denoyer says. "Highly inert deactivation technologies along with increasingly sensitive detectors are major advances that ensure an inert flow path consisting of deactivated inlets, liners, and columns."

Quadrupole GC-MS System
TSQ 8000 | Thermo Fisher Scientific
www.thermoscientific.com

INSIGHTS ON GAS CHROMATOGRAPHY SYSTEMS

THE HEART OF THE MATTER

▲ *Column Selection Tool | GC Column Configurator | Thermo Fisher Scientific | www.thermoscientific.com*

▲ *Gas Generator | NitroFlow 60 Parker Balston | www.parker.com*

▲ *5 % Phenyl GC Column Range BP5MS | SGE Analytical Science www.sge.com*

Capillary columns have changed the face of GC since their introduction about 35 years ago. The most obvious change involves resolving power: up to 50,000 theoretical plates on a 30-m capillary versus 1500 on a six-foot packed column. Capillary columns also permit the rapid heating and cooling that is one hallmark of "fast GC." Packed columns are still used, however, for high-volume injections and gases in particular or when expert chromatographers wish to experiment with stationary phases.

Major GC system and column manufacturers still emphasize innovation in column technology. "Everyone still pays close attention to the stationary phases, to match column phases to compound classes and reduce bleed," says Eric Phillips, GC and GC-MS marketing manager at Thermo Fisher Scientific (San Jose, CA). "Column chemistries just keep getting better."

GC column performance depends on the complex interactions among several factors: column length and diameter, chromatographic conditions like mobile phase flow and temperature, and chemical interactions between analytes and stationary phase.

Selecting a GC column begins with the organic chemistry maxim that like dissolves like. "You should target selectivity first," advises Chris English, who manages Restek's (Bellefonte, PA) Innovations Laboratory. "If you're dealing with glycols, select a phase that's most like a glycol. If you're analyzing gasoline, select a nonpolar phase like polydimethylsiloxane." Stationary phases that dissolve analytes provide the optimal retention, assuming optimization of the remaining conditions.

Obtaining acceptable retention is also possible by using a less-than-optimal stationary phase in a longer column. Doubling the column length adds approximately 40 percent theoretical improvement in resolution, but the column will cost twice as much and may not last as long as a shorter column.

Similarly, narrower-bore columns resolve analytes more efficiently but require much higher back pressures. Since column capacity is roughly related to the square of column diameter (as with cylinders), loading capacity falls off dramatically from 1 mm to 0.18 or 0.10 mm. This can significantly compress the concentration calibration curve, thus limiting applicable concentration ranges.

Longer columns provide greater efficiency and resolution but at the expense of longer retention times. Thicker stationary phases also improve retention, especially for volatile compounds, but suffer from higher bleed and slightly lower efficiencies.

DELICATE BALANCE

Expert chromatographers achieve the separations they desire by balancing the column's physical and chemical characteristics. Approximately the same advantage in resolution is gained by switching from a 0.25 mm ID column to 0.18 mm. It is therefore possible to trade off the wider bore for a shorter column and reach approximately the same column efficiency. Similarly one could, at 0.18 mm or even 0.10 mm ID,

increase the film thickness to compensate for the lower capacity of narrow-bore capillaries. This will change the number of theoretical plates as well but will maintain to a large degree the required calibration curves.

Phenomenex (Torrance, CA) has an hour-long presentation on its website on selecting a GC column. Kory Kelly, GC product manager offers this tip: "Know your analytes. The most efficient separations depend on differences in their chemical and physical properties."

GC separates materials based on two properties: boiling point and chemical interactions with the stationary phase. Elution based on boiling point differences is straightforward enough. Those with lower boiling points vaporize and elute first. The wider the boiling point differences, the more complete the separation.

"Column chemistries just keep getting better."

For compounds with very similar boiling points, chromatographers must rely on retention differences based on interactions with the stationary phase. Here the idea of "like dissolves like" comes into play. With a binary mixture of one very polar and one weakly polar compound, a polar column would retain the more polar material more efficiently, while a hydrocarbon-like column would more efficiently retain the less-polar compound.

It's not quite that simple, however, as the temperature stabilities of the analytes and the column must be considered. This same binary mixture will not separate on highly polar columns with an upper temperature limit of 250° C if one analyte boils at 400° C. "Clearly, maximum column temperature is a fundamental limitation. The strengths and types of interaction between the analytes and stationary phase must be balanced against that number," Kelly says. Generally speaking, the more strongly a column interacts with analytes (i.e., the more polar the stationary phase), the lower its temperature stability. Highly polar polyethylene glycol stationary phases are stable to about 260° C, whereas the highly nonpolar polydimethylsiloxane is highly temperature-stable.

Analyte stability, while it does not affect the column, may directly or indirectly influence column selection. Highly functionalized molecules of even low molecular weight, for example amino acids, must be derivatized or "capped" to survive GC conditions.

"The goal is to optimize differences in chemical interactions between analytes and the stationary phase," Kelly notes. This strategy sometimes leads to methods that violate the "like dissolves like" rule. For example 1,2 dimethyl benzene (xylene) easily separates from a nonaromatic hydrocarbon of identical molecular weight. However mixtures of 1,2; 1,3; and 1,4 xylenes have identical molecular weights and aromaticity, have nearly identical boiling points, and will co-elute on a 50 percent phenyl column. However, a very polar cyano or polyethylene glycol stationary phase will induce dipoles in the molecules sufficient to enable clean separation.

HYDROGEN OR HELIUM?

The recent helium shortage has created opportunities for cost-effective, alternative carrier gases, but the significance of the switch is by no means settled.

On August 11, 2012, the *Wall Street Journal* reported, due to a legal quirk, the imminent closure of the Federal Helium Reserve, a profitable helium-producing plant initially supported by federal funds. Congress has introduced legislation to keep the plant open, but its future remains in doubt and prices are likely to remain high.

Phil Allison, manager of sales and marketing at Parker Hannifin (Fairfield, NJ) makes a compelling argument for hydrogen generators replacing helium tanks, based on the newer technology's cost-effectiveness and convenience.

"The helium shortage became a buzz a few years ago; then died out, then returned," Allison says. "Over the past six months we've seen a definite uptick in chromatographers seeking alternatives to helium cylinders."

Cost and convenience are major drivers. Allison reports that one of his customers used to go through six helium cylinders per week, at a cost of $345 each. "That puts the economic advantages of on-site hydrogen generation into perspective." The shortage is so dire, Allison says, that consumer markets (e.g., party stores) are feeling the pinch. "Clearly the tight market is causing rationing or at best is forcing users to settle for lower-grade gas."

Price is just one component of the cost of tanked gases, Allison says. Indirect costs like cylinder rental, delivery fees, administrative costs, tank management, wastage (only 90 to 95 percent of tanked gas is accessible), inventorying, and complying with codes and standards add approximately $100 to the cost per cylinder.

That is why Parker Hannifin views replacing helium tanks with hydrogen cylinders as a halfway solution. Tanks are cumbersome, costly to transport and rent, and dangerous due to their sheer weight.

Parker Hannifin specializes in on-site generators that use a palladium membrane to produce high-quality hydrogen through water electrolysis. According to Allison, generators can pay for themselves in about one year, but ROI varies depending on volumes.

"Perhaps the best argument against switching to hydrogen involves validated methods."

At just one-fourth the density of helium and one-twenty-eighth that of nitrogen, hydrogen travels much faster through capillary columns and spares the column by permitting lower-temperature elution. Allison claims hydrogen also provides greater sensitivity at the detector end and requires no retrofitting or modification to recent-vintage instruments. "Almost every GC manufactured today has the capacity to use hydrogen carrier gas."

Hydrogen has suffered a bad reputation since the Hindenburg disaster in 1937. The tiniest spark causes hydrogen to combine explosively with atmospheric oxygen. Yet generators create hydrogen on demand, so the buildup of dangerously large volumes is impossible. The plumbing between generator and instrument is protected against potential sources of sparks, and built-in electronic pressure control shuts generators down if the gas line is compromised.

"Hydrogen has the benefit of providing better resolution than helium, which creates the opportunity to speed up GC analysis," says Cynthia Cai, Agilent gas phase solutions and commercialization manager. It also frees chromatographers from the vagaries of the specialty gas marketplace, especially when a hydrogen generator replaces hydrogen cylinders. But Cai notes that "using hydrogen as a carrier gas requires additional safety precautions," and the switch from more common carrier gases will likely affect methods.

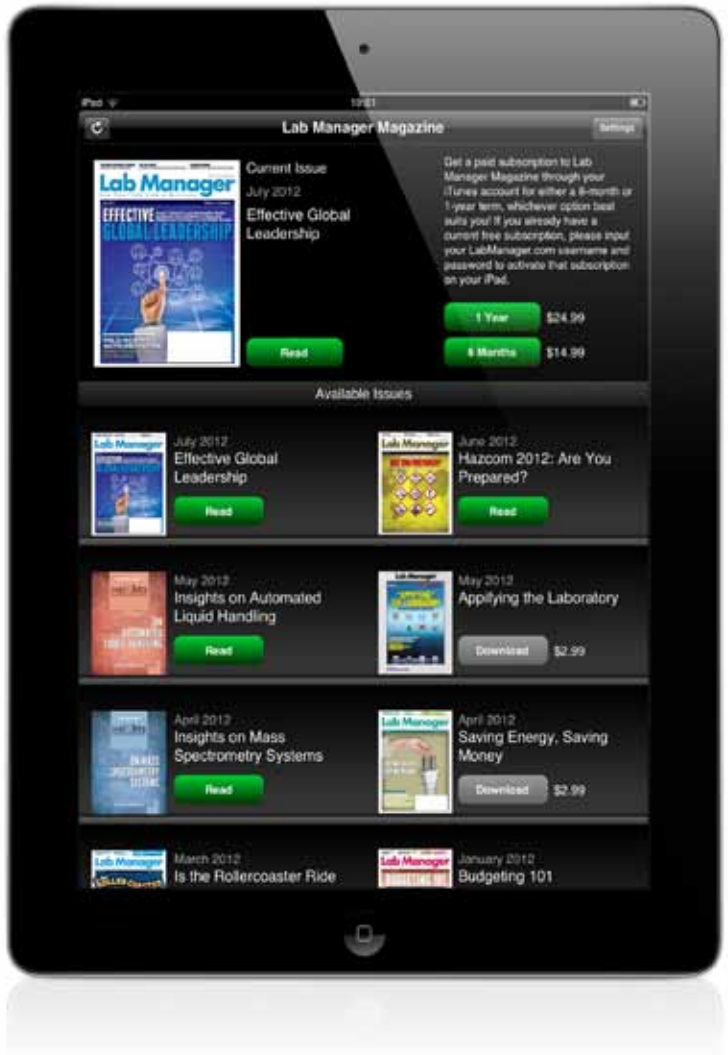
Not everyone agrees that hydrogen is a panacea. An August 27 article in *Forbes* online indicated that shortages are due to temporary events like pipelines and production facilities closing for routine maintenance. According to the U.S. Geological Survey, the world's helium consumption is 180 million cubic meters but reserves are estimated at 50 billion cubic meters—a 300-year supply. Moreover new sources are coming online as a side-product of shale gas extraction (fracking).

Fran Kandl, product manager for specialty gas equipment at Airgas (Allentown, PA), is among the helium shortage skeptics. While acknowledging that prices have risen and that hydrogen as a carrier gas can be beneficial in some circumstances, he cites overusage and waste as factors that cloud the actual cost of carrier gas helium. "In most cases, the helium consumed for [non-GC] lab applications is much higher than what the GCs actually use. A GC running continuously consumes between 20 and 40 cubic feet per month, so one 300-cubic-foot cylinder can run five to six instruments for a month."

Waste includes overuse of carrier gas, fuel gas, gas vented off the split-purge, and makeup gases, plus leaks. Because of hydrogen's lower density and molecular weight, the cumulative effects of leaks are magnified relative to helium. Airgas provides customers with a gas flow calculator that quantifies potential sources of waste and a program that teaches customers how to conserve gas by making modest modifications to the gas delivery system and usage practices.

Perhaps the best argument against switching to hydrogen involves validated methods. Many labs, particularly in pharmaceutical, environmental, and forensics, employ methods from FDA, EPA, ISO, etc., that may require validation of every peak. The helium-to-hydrogen switch would entail rewriting and revalidating methods—a costly, time-consuming exercise.

"The helium shortage provides the incentive for us to work with our customers to dramatically reduce their usage of carrier gases," Kandl says. "Even with the shortage, it's possible to save 30 percent without even trying very hard."



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INSIGHTS ON GAS CHROMATOGRAPHY SYSTEMS

IT COMES DOWN TO SPECIFICITY AND AVAILABLE SAMPLE PREP TIME

Thermo Fisher's Eric Phillips, describes the adoption of MS detectors in GC as a "technology shift" that began with single-quad MS as an alternative to standard GC detection modes. "Now a lot of single-quad work is yielding to GC triple quad because of the latter's capabilities and dramatic price reductions," Phillips explains.

Phillips concedes that detectors using flame ionization, electron capture, and nitrogen/phosphorous detection "do fantastic work" and still constitute approximately three-fourths of all GC detector sales. But conventional GC detectors fall short of MS's confirmation of molecular identity through precise mass measurement.

High performance comes at a cost, however. A GC with a single conventional detector, autosampler, and data system costs between $20,000 and $25,000. With a single-quad or ion trap, the price reaches the low $70,000s, and the price runs $120,000 for an entry-level triple quad and $150,000 for a top-of-the-line configuration.

The business case for MS detection matters less about the industry than it does the specificity required to get the job done. "Switching from a non-MS detector to a single quad comes down to how specific you need to be, and how much time you have available for sample prep," Phillips explains. As MS detectors increase in sophistication, so does their ability to resolve co-eluting peaks and analytes from matrix within the ionization chamber, a process known as infusion.

Phillips describes MS as a "universal detector" that is simultaneously highly specific. "There are times you don't need it, which is why labs still use non-MS detectors. But when you need it, you need it."

▲ *BiPolar MALDI TOF Detector PHOTONIS USA | www.photonis.com*

"Upgrading to MS detection will increase the instrument's sensitivity and selectivity."

▲ *Pulsed Discharge Detector | VICI D-3-1-HP / Valco Instruments www.vici.com*

According to Phillips, MS paradoxically requires less experience to run expertly despite higher instrument complexity. "Someone new to the field can be trained on GC-MS and be positive they're locating their target compounds. Conventional GC detectors require more experience to understand when problems arise or when matching peaks to known retention times. MS avoids a lot of those problems."

Workflows are similarly enhanced by MS's higher efficiency, at least for some applications. "The more specificity you obtain for your list of compounds, the easier the analysis. That, combined with software that points out areas failing a QC specification or known problem areas, can speed things up. Not the chromatography, but the ability to analyze or view data and confirm results."

According to Erik Hansen, VP of commercial operations at IONICS Mass Spectrometry (Ontario, Canada), upgrading to MS detection will increase the instrument's sensitivity and selectivity and thereby, at least in the case of triple quads, improve the lowest level of quantitation. "Sensitivity improvements allow labs to refine assays that detect at levels unavailable by other means."

"MS is the standard way to attack complex mixtures that include unknowns," comments Jack Driscoll, technology and marketing manager at PID Analyzers (Sandwich, MA), adding, "particularly if you have $60,000. But MS is overkill for many applications like QA/QC labs that repeatedly target a limited number of analytes."

"MS paradoxically requires less experience to run expertly despite higher instrument complexity."

"You can spend between $4,000 and $7,000 on a non-MS detector and get the job done, often at higher sensitivity."

According to Driscoll, a photoionization detector is "much more sensitive" than MS is for aromatic compounds, achieving lower ppb detection limits.

Lower-cost non-MS detectors more readily (and cheaply) allow dual detection, such as photoionization plus flame ionization. This particular combination detects unsaturation (PID) and "everything" (FID). The PID/FID ratio quantifies a sample's olefinic content and is prescribed by many state environmental and EPA methods for hydrocarbon analysis. "The combined detectors, which cost about $8,000, give the same results as running two separate columns in series."

MS Detector | Flexar SQ 300 PerkinElmer
www.perkinelmer.com

INSIGHTS ON GAS CHROMATOGRAPHY SYSTEMS

OPTIONS RANGE FROM OEM SERVICE SUPPORT TO DO-IT-YOURSELF

The proverbial "ounce of prevention" goes a long way toward preventing serious GC downtime. Keeping up with routine maintenance is the secret to ensuring that scheduled maintenance downtime occurs on the lab's terms, not by fickle fate. The major instrument makers, says Thermo Fisher's Eric Phillips, make a big deal about what his company calls "robustness"—the length of time between cleanings—which "has a tremendous impact on productivity. Nobody wants an instrument to go down because of maintenance or contamination at inopportune times."

The hierarchy of service support begins with the OEM and proceeds through a large national third-party service organization, local service engineering firms, and mom-and-pop shops. The OEM is always the first line of defense for service, but many customers are dissatisfied with OEM response time or pricing. Larger vendors such as PerkinElmer provide service techs who handle their competitors as well as their own chromatographs. Through the firm's OneSource service, PerkinElmer instrument specialists work full-time on-site, available to problem-solve most instrument issues. For instruments for which they lack expertise, they arrange for service by either the original vendor or a third-party service provider.

▲ *Gas Chromatography Columns AbelBonded® | Abel Industries www.abel-industries.com*

> "It's normally not the analyte of interest that causes column problems, but the matrix."

Injection port maintenance is one of the simplest services users can perform. Almost any operator can change the septum. A bit more skill is required to service the port liner or clean out the port itself.

"There's a fuzzy line between user-serviceable fixes and calling for service," notes Brian Lewandowski, implementation specialist at PerkinElmer (Waltham, MA). "It depends on the user's comfort level for carrying out specific maintenance tasks. The button-pushers will call a technician at the drop of a hat. Those who have been around chromatography for a while lean toward solving problems themselves." A service visit takes at least 24 hours; user-initiated service is much faster and far less costly when hourly rate and lack of productivity are factored in.

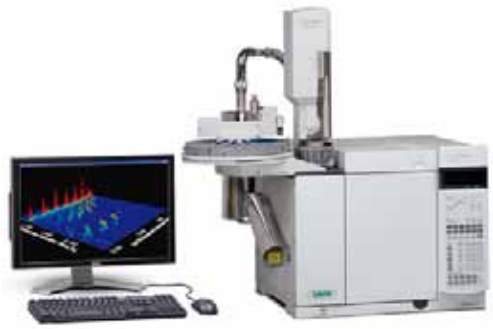
Injection port maintenance is one of the keys to keeping columns in good operating order. The other—although not strictly maintenance—is exercising care in sample preparation. All columns eventually degrade. Aside from adding a guard column and snipping off a foot or so, little can be done to "service" a column. This was as true in the days of quarter-inch packed columns as it is today with 0.1 mm capillaries.

▲ *GC-MS System | GCMS-TQ8030 Shimadzu | www.ssi.shimadzu.com*

While columns cannot be fixed, users can follow a few recommendations to improve their longevity and performance. "It comes down to how the column is stored, how it's handled while in the GC, its temperature experience, and what's shot through it," Lewandowski advises. "It's normally not the analyte of interest that causes column problems, but the matrix, such as sludge in petroleum products, or water, that cause column problems."

"Those who have been around chromatography for a while lean toward solving problems themselves."

Although some users can swap out a circuit board or major component, hardware failure and electronics glitches almost always require service engineers. Some instruments, such as the Agilent 5890, are no longer supported by the manufacturer and can be serviced only by third-party service organizations. Because tens of thousands of 5890s are still in service, a few users have learned to service them, provided they can find spare parts. The same cannot be said for very recent instrument releases.



GCxGC System | LECO
www.leco.com

INSIGHTS ON GAS CHROMATOGRAPHY SYSTEMS

BRINGING THE LAB TO THE PLANT

Process GC involves the deployment of rugged, reliable gas chromatographs in demanding process environments. Where traditional sampling and analysis occurs off-site in analytical laboratories, process GC brings the "lab" to the production site, providing real-time product analysis.

Although most common in oil and gas industries, process GC is slowly entering other process markets. Al Kania, GC product manager for North America at Siemens (Houston, TX), estimates that such esoteric measurements as acetic acid in ketchup, alcohol in whiskey, aerospace materials analysis, monitoring the destruction of nerve gases, and others may compose two percent of sales.

Although laboratory and process GCs are based on the same principles, significant differences exist. Where lab analysis can take half an hour or more, process analytics are quite rapid—most being over in a few minutes. "For decades, process GC has been using multidimensional analysis to speed things up," Kania observes. Multidimensional GC, only now catching on for lab applications, involves autoinjection of samples onto one column, separating and backflushing matrix or background components, and rerouting the analytes to as many as seven different columns.

> "For decades, process GC has been using multidimensional analysis to speed things up."

Like QA/QC chromatographs, process GCs operate nearly continuously and tend to be dedicated to picking out specific analytes. Pure research GCs are much more flexible with respect to operation and detection mode.

Perhaps the most striking difference is the ruggedness of process instruments. GCs mounted in metal sheds in the middle of a refinery experience temperature extremes and explosive gases, which seriously restrict their electronics and detector choices. "Lab instruments exist in more of an office environment," Kania says.

Process GCs cannot use high-voltage pulsed-discharge detectors because of the potential for explosions. And since they operate unattended, detectors must be extremely stable. Only a handful of detectors fit the bill: thermal conductivity, flame ionization, and flame photometric detectors are most common; photoionization or electron capture detectors less so. Even these must be mounted in explosion-proof steel blocks.

▲ *Industrial GC with FID & TCD*
Buck Scientific | www.bucksci.com

Ruggedness extends to reliability as well. Even today labs usually have one or two individuals who perform routine maintenance and diagnostics. Refinery engineers have little experience in instrument operation and care. Process GCs must therefore be self-correcting and self-diagnosing, and components must be plug-and-play.

Despite obvious differences, there is significant crossover between lab and process instrumentation. Wasson ECE Instrumentation (Fort Collins, CO) is one company that repackages and ruggedizes laboratory instrumentation for process environments, particularly those with unusual detector requirements. These instruments will not be as rugged or reliable as much more costly process instruments, but they serve niche markets.

Conversely, some process industries with widely dispersed points of production will use automated sample collection in the field and bring the samples to a continuously operating process GC located in a lab.

By no means have process GCs made lab instruments obsolete in their industries. "Workers still take physical samples, and at the end of the day, laboratory GC is still employed for spot tests, for troubleshooting, and to validate answers from process GCs," Kania tells *Lab Manager Magazine*. "Lab instruments remain the gold standard, the stamp of approval, for product release."

Micro Process Gas Chromatograph Sitrans CV | Siemens Industry www.usa.siemens.com

Q&A WITH SELECT GAS CHROMATOGRAPHY EXPERTS

OUR EXPERTS:

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Naugatuck, CT

Gary Deger
Marketing Director
CDS Analytical
Oxford, PA

Daniel Fabry
Instrument Specialist
Haverford College
Haverford, PA

Philip J Marriott, Ph.D.
Professor of Chemistry
Monash University, Clayton
Victoria, Australia

William Terzaghi, Ph.D.
Professor of Biology
Wilkes University
Wilkes-Barre, PA

Q: Describe your organization and how it uses GC.

A: **Andrew Skroly:** CDS is a specialty chemical company supplying the automotive, aerospace, refrigerant, and polymers industries. We use GC every day to characterize new products, support our R&D functions, troubleshoot customer complaints, and assay raw ingredients for antioxidants, polyol esters, solvents, urethane prepolymers, and others.

Gary Deger: We manufacture GC injection solutions and so run test samples for prospective customers and for studying new applications. Specifically, we make systems that eliminate GC sample prep. We generally analyze polymers, and our systems are used almost daily.

Daniel Fabry: Haverford is a small liberal arts college with a strong research-based chemistry program. We use GC-MS to identify or confirm molecular weights and GC-FID to monitor the progress of chemical reactions. At Haverford, we use GC within our organic and environmental research labs. The organic group studies natural products for potential medicinal applications; the environmental group analyzes samples from the Gulf of Mexico.

Philip Marriott: As an academic institution, we use GC to develop analytical methods in comprehensive two-dimensional gas chromatography (GCxGC) and multidimensional gas chromatography (MDGC), usually supported by MS detection. Our published research includes fundamental relationships in advanced GC methods, method developments for high-resolution chemical separations, and applications of GC, MDGC, GCxGC to demonstrate the scope and applicability of our methods to complex samples. Our samples include petrochemicals, pesticides, fatty acids, essential oils, aroma compounds, flavonoids and polyphenols, and illicit drugs.

William Terzaghi: My group primarily employs GC to analyze resveratrol content of various plant tissues and for characterizing fatty acids and lipids in plant tissues. Although we share the instrument with other groups, we utilize it at least one full day per week.

Q: What kinds of detectors do you use, and why?

A: **Andrew Skroly:** We use MS, FID, and TCD. Together, they provide a multitude of options for the many different samples we are asked to analyze.

Gary Deger: We use only MS detectors. Most of our samples are analyzed for unknown polymers and additives in plastics, rubbers, coatings,

biofuel source material, and tobacco, among others.

Daniel Fabry: Haverford has three instruments: a Perkin Elmer Clarus GC-MS, an Agilent 7890 outfitted with a FID and MS detector, and a new Shimadzu-2014 GC with an FID. The Agilent 7890 is a dual inlet system with a PTV inlet; the Shimadzu GC is used to monitor reaction progress with mostly isothermal temperature programs. We employ the PerkinElmer GC-MS for identification and confirmation. We employ a variety of temperature programs depending on the column type and compound of interest.

Philip Marriott: We use a variety of detectors: a nitrogen-phosphorous detector for atmospheric samples, petrochemicals, and smoke; a flame photometric detector (FPD) for sulfur compounds from varied samples, FPD/P mode for organophosphate pesticides, phosphate esters in flame retardants, and phosphorous in chemical weapons; electron capture for chlorinated pesticides and biphenyls; olfactometry (sniffing detection) for aroma compounds in wine, herbs, spices, and coffee; FID, quadrupole MS for general applications; and time-of-flight MS for GCxGC.

William Terzaghi: We use MS detection for all of our samples [in order] to obtain positive identification of every peak.

Q: What are the most significant bottlenecks in your GC workflows, and how do you overcome them?

A: **Andrew Skroly:** There are not many bottlenecks in our workflows. We have five GCs in the lab that we have set up for nonpolar, wax, and polar columns. Our injectors consist of on-column to split/splitless.

Gary Deger: We really do not have any workflow issues unless a system is down, which can result in samples backing up. We try to fix as many problems as we can ourselves, and if we cannot, then we call in a GC service engineer. Sometimes there is a small delay in getting them in, but never more than a few days. If anything, I would complain about their rates being too high.

Daniel Fabry: The most significant roadblocks arise from oven cooling after a high-temperature program ramp. At the forensic lab, a major bottleneck has been instrument and host computer communication. Recently, our Agilent GC-FID/MS has had some leak issues originating from our inlet setup. Of course, maintenance is always a bottleneck but that cannot be avoided. I perform instrument maintenance regularly to avoid any large issues from lack of care and cleaning. Instrument computers often generate problems when trying to connect to a network or upgrade software. Most instruments require a hardware upgrade before you can run them with a different operating system, which can be costly, and Microsoft Updates can be a source of problems if your computer is networked. Bottlenecks in workflow are most often remedied by good communication, proper care, and a good support system that can help you when things go wrong.

Philip Marriott: Since our work is exclusively basic GC research rather than routine applications involving classical workflow considerations, we are continually changing methods, changing columns, and reconfiguring our GCs for advanced, multicolumn methods. Our GC workflow normally includes activities such as Deans switch balancing, entering complex procedures for event control, then testing the method for reliability and performance. We also require very fast performance of all our detectors, so the MS acquisition rate is critical.

William Terzaghi: Our most significant bottlenecks are sample throughput and instrument availability. Once we have acquired sufficient preliminary data, we hope to write a grant to purchase a GC-MS exclusively devoted to our projects [in order] to improve the availability, and we are always looking for ways to shorten the turnaround time between samples by altering programs.

Q: What can GC systems vendors do to improve their products and/or streamline your workflow?

A: **Andrew Skroly:** I'd like to see systems where you could change out detectors depending upon the analytes in question. Another improvement would be better communication with LIMSs, including improved ability to dump data and operate the GC.

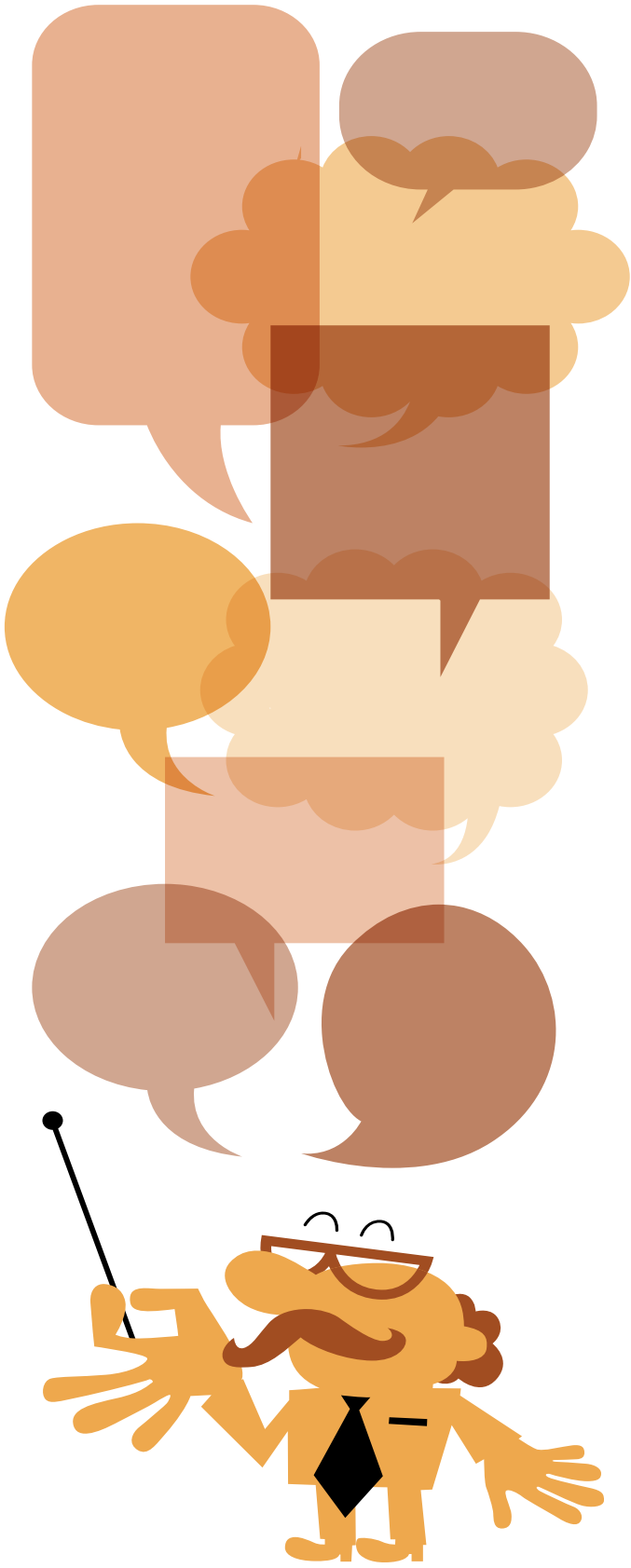
Gary Deger: Most GC-MS systems perform similarly, but the operating software can be an issue. We have systems from all the major vendors, but some

of the user interfaces crash or lock up more than others [do].

Daniel Fabry: There are two aspects to acquiring an instrument: the instrument itself and service. The major vendors should hire more service engineers who cover smaller areas, regardless of whether customers purchase service contracts. There is one major vendor, in particular, that has significant room for improvement in this regard. Agilent has started a YouTube channel that offers troubleshooting and maintenance tips that have been a huge help. Haverford College employs me to fix and maintain their instrumentation and does not purchase maintenance contracts. Every company treats their noncontract clients differently, but I feel that more companies are choosing to hire in-house instrument specialists to avoid the high cost of instrument contracts. I feel response time and willingness to divulge technical information varies for contract and noncontract customers. Another way GC vendors can improve my workflow is [by] offering a functional website for finding consumables. I cannot tell you how many times I enter a product number in a search and nothing comes up.

Philip Marriott: We have an interest in making MDGC methods setup more systematic, with method development guidelines or simulations that can provide accurate balancing conditions, and improvement in automated entries into events tables via a GUI interface. Generally, faster MS would be of advantage, especially for high-resolution TOF-MS. In terms of GCxGC, there is a continuing need for innovation in the areas of software for data acquisition, presentation, and interpretation. These improvements would help GC and GC-MS users with new capabilities, especially if the future of GC becomes more directed to MDGC and GCxGC technology.

William Terzaghi: Vendors can make machines that shorten the run time for each sample. It will also be useful if they could develop kits for sample preparation that would shorten the sample prep time. Finally, columns that allow us to resolve resveratrol and its glycosylated forms without derivatization would be very helpful.



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