ADVANCING GC METHODS AND ENVIRONMENTAL ANALYSIS
Gas chromatography (GC) is a powerful technique for environmental analysis. The articles in this new e-book look at both the technique and its application, providing essential information about GC fundamentals as well as an example of how the technique can be used in new ways in environmental analysis.

First, Raquel Fernández Varela talks about her use of gas chromatography–mass spectrometry (GC–MS) for the analysis of marine pollution. She used GC–MS to study how marine polychaetes respond to contamination from oil spills. Her method had to address important challenges related to the wide range of sample compounds involved as well as complex data analysis.

Next, John Hinshaw presents a “GC Troubleshooter’s Toolkit.” This guide to the essential tools for installing, maintaining, and troubleshooting GC instruments and columns not only explains the proper use of traditional laboratory tools, but also offers some surprising suggestions for using unconventional items in the GC lab, including some you never think of as “tools” at all.

Our last piece provides practical information about how to choose the right GC column for a given analysis. The article provides five overall selection rules, as well as advice about choosing physical column dimensions such as length, internal diameter, and film thickness. It also reviews the key analyte and stationary-phase interactions that must be considered.

We hope you enjoy this e-book, and find it helpful in your work in environmental analysis.
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Marine polychaetes are a common type of annelid worm widely spread in marine environments. In this interview, Raquel Fernández of the University of Copenhagen, Denmark, talks about her innovative approach to developing an untargeted method to monitor polychaetes and to assess their potential use in environmental monitoring of oil spills.

You recently developed a gas chromatography–mass spectrometry (GC–MS) metabolomics platform to investigate marine polychaetes.

Why are you investigating marine polychaetes?

The idea arose after my PhD, which dealt with monitoring and characterizing the oil spill that resulted from the 2002 Prestige oil tanker disaster off the coast of Galicia, in northwest Spain. I then applied for an IEF Marie Curie Fellowship with Prof. J.H. Christensen at the University of Copenhagen (Copenhagen, Denmark).

One of the requirements of the fellowship was that the project had to be innovative, and despite the fact that marine polychaetes are ubiquitous in the marine environment, no studies had been performed to investigate the potential of these organisms for biological...
monitoring. My objective was to examine how hydrocarbon pollution and other environmental stressors control the metabolic response at a molecular level and evaluate their use as pollution indicators. If a response could be determined, the limited mobility and tolerance to polluted conditions of marine polychaetes would make them an excellent candidate for biological monitoring.

What were the limitations of existing analytical tools when developing this method?
The main limitation was that there was no analytical platform that could cover all the compounds. This was, and still is, especially true for untargeted studies in which the interest can span multiple classes of metabolites. When the study began we looked at the existing analytical platforms and after an initial screening, we first thought that ultrahigh-performance liquid chromatography–mass spectrometry (UHPLC–MS) would be the best candidate for our purposes. However, the data analysis and the preprocessing turned out to be especially problematic because the shift pattern in retention time and sheer number of samples proved to be too complex a problem to be adequately corrected using the bioinformatics tools we had at the time. Hence, we decided to move from liquid chromatography (LC) to gas chromatography (GC), because GC tends to be more stable in terms of retention time. This implied that the separation criteria and targeted class of compounds of primary interest also shifted from more water-soluble to more hydrophobic compounds. The unsuitability of GC for nonvolatile or thermally unstable compounds also required a suitable derivatization scheme before we could proceed with the GC analysis.

What is novel about your approach? And what challenges did you have to overcome from an analytical perspective?
The most novel aspect is that we presented an entire method for environmental metabolomics in marine worms from sample preparation and extraction, to chemical analysis, data preprocessing, and multivariate methods. We included all the steps needed to perform a good untargeted analysis. One, if not the biggest, challenge we had to face was the optimization of the parameters for the preprocessing using XCMS. XCMS is free open-source software to process metabolomics data that includes feature detection, retention time alignment, and feature matching across samples to generate a consensus table of features common to a majority of the samples. The package is mature and it has been successfully used in many metabolomics studies. This software suite was developed initially to preprocess LC–MS-based data, and to our knowledge, it is used predominantly for this purpose. However, with some optimization to the
parameters, it can be successfully used for treating GC–MS-based metabolomics data, as we show in our approach. We worked for days on testing the different algorithms available in XCMS — for feature detection, peak matching, and retention time alignment; different combinations of parameters; and even developed in-house a number of additional software tools and diagnostics to check the results — but yet, we could not find a combination that worked for all the samples at once and satisfied us entirely. In fact, the difficulties in preprocessing were the main reason why we shifted to GC in the first place.

What were your main findings?
The main finding was that a specific metabolite pattern variation in marine polychaetes could be correlated with oil exposure, meaning that metabolite patterns could serve as oil pollution indicators in the future. However, no biological variation was taken into account in this study and only the remaining data we collected during the project could throw some light on this. Another result that I really think is important, though it might not appear as such at first glance, is that we really understood the importance of the incorporation of quality control (QC) samples (for example, a pooled sample) not only for the chemical analysis, but also for the quality of the preprocessing, to reduce analytical variation and to quantitatively determine analytical precision. Finally, we showed how the normalization of the analytical profiles is essential to the proper profiling of metabolic data; we compared three different approaches to investigate which one is the most suitable for our particular case.

Are there any specific problems encountered when developing an untargeted GC–MS method?
The main problem in untargeted GC–MS metabolomics is achieving a well-resolved chromatographic separation of a large number of metabolite species with different chemical and physical properties in a single analytical run before performing the data preprocessing. Also, the very heterogeneity of the data made the optimization and validation of the parameters for the data preprocessing especially difficult, yet essential to the whole method. And, again, it was only by using the QC samples that we achieved our results. The comparison of the XCMS results against manual integration of selected peaks relative to different classes of compounds showed an excellent correlation. It is evident that XCMS offers optimal choice of parameters to provide an accurate feature extraction, comparable with manual peak integration.

Is there any general advice you would offer to chromatographers attempting to develop untargeted methods?
One of the biggest issues in metabolomics is reproducibility. Often the technical variation is high, despite
the fact that no biological variation could be observed in this study. We managed to show the need to include a large number of QC samples in each batch or sequence. Even though many have pointed this out, I believe this cannot be stressed enough! Another important point is not to underestimate the power and the effect of proper data preprocessing. Also, in general, it can be extremely time consuming. For space reasons, it may not take too much space in published works, and it often looks simpler and easier than it actually is. It is not “point-and-click”! It requires knowledge about your samples, right-thinking choices, and awareness of the consequences it may have on the data analysis. In this respect, I think, the third and most important point is this: Always go back to the original data and double-check that what you get from the data analysis shows in the raw data.

Are you developing this project further? I believe that the method we developed can be used in real-life studies. This study was limited by the experimental design, which did not allow us to distinguish biological variation. We have additional data where this aspect was taken into account; these data seem to show that we could separate marine polychaetes exposed to different levels of contamination. The data analysis though is not complete and we need to thoroughly validate the results.

The plan is to write them into one or two additional papers, but my current involvement in other projects in microbial metabolomics at Chr Hansen A/S (Denmark) makes it difficult to do it right now.

Raquel Fernández Varela has a M.Sc. and a Ph.D. degree in analytical chemistry, both from University of A Coruña (Spain). She moved to Denmark in 2011 with a Marie Curie Intra European Fellowship to performed postdoctoral studies at the Department of Plant and Environmental Sciences at the University of Copenhagen (Denmark). Her main research at that point was to examine how pollution and other environmental stressors control metabolite response in selected organisms at the molecular level. In September 2014 she moved to Chr Hansen A/S (Denmark), where she is currently working as a research scientist in microbial metabolomics. She has extensive experience in gas chromatography, mass spectrometry, applied multivariate methods, and environmental and microbial metabolomics.

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Here, we present tools and accessories that gas chromatographers use in the laboratory.

Some years have passed since this list was last published in LCGC (1). During the interim, my toolbox has gained some new items, and a few others have seen little or no use. Given the modern obsession with list-making, readers may enjoy this update and perhaps learn about some tools they might like to add or replace in their own toolkits.

Every profession has its specialized tools. Those used in chromatography often are just as specialized as those used in computer repair or automotive work. Many of the tools and accessories that gas chromatographers keep on hand for installing, maintaining, and repairing their chromatographs are also found in the toolkits of plumbers, carpenters, and homeowners. Wrenches, screwdrivers, pliers, and metal-tubing cutters are some easily recognized examples. Other items such as dental instruments or paper correction fluid are familiar, but their use in the laboratory environment might not be immediately obvious. Still others, like a column flowmeter, septum nut wrench, or a specialized fused-silica column cutter, aren’t found outside the laboratory at all.
Here, then, is the list of today’s tools and accessories along with some information about their use and significance. One or more specialty manufacturers offer many of these chromatography-specific items in their catalogs or on-line offerings. I scanned through several catalogs and web sites and gleaned some new items that I have included here.

**Butane Lighter**
A butane lighter is a convenient source of hydrocarbon gas for measuring an approximate unretained peak time. Butane is effectively unretained at temperatures above 75 °C on liquid-phase coated columns with phase ratios above 50. Columns at low temperatures or with lower phase ratios (thick stationary films) may retain butane and separate the traces of ethane and propane present in the butane fuel. Use the earliest observable peak for the best estimate of unretained peak time. Natural gas is mostly methane; if your laboratory has a supply of natural gas (mine doesn’t) it makes a good substitute for a lighter and is less retained than butane. Just be sure to turn off the gas after you’ve filled a syringe with it. A lecture bottle of methane with a suitable pressure regulator is another excellent source of the unretained substance. Concentrations in the low percent range work well. Hydrocarbons won’t work for unretained peak time measurements with electron-capture detection (ECD). Instead, try loading the syringe with a puff from a pressurized can of dust remover such as Dust-Off, which contains 100% 1,1-difluoroethane.

Hydrogen or helium—whichever is not the same as the carrier gas—make good unretained peak markers for porous polymer or molecular sieve columns that retain hydrocarbons strongly, plus these two gases should be readily available in most gas chromatography (GC) laboratories. Flame ionization detection (FID) will not respond to hydrogen or helium, but other detection methods such as thermal conductivity detection (TCD), pulsed discharge detection (PDD), or helium ionization detection (HID), the latter with hydrogen as the unretained substance, should respond well.

**Cable, Three-Wire**
Recorder cable finally has gone the way of chart recorders: You might find some lying around but they haven’t been used in years. I have one or two in the lab just in case the need arises. I keep some spare USB cables as well.

**Correction Fluid**
Use white correction fluid to mark the measured position on a column that corresponds to the correct column penetration depth into an inlet or detector. Measure the depth after inserting the column into the nut and ferrule and making a fresh cut on the column end. Some regulated laboratories’ policies don’t permit the use of correction
Cutters, Fused-Silica Column
The best fused-silica column cutting tool is the one that holds the column in an adjustable chuck and cuts with a diamond chip as the operator rotates a thumb wheel. This tool also has a magnifying glass on the opposite end for inspecting the fresh cut for squareness and lack of burrs or hanging polyimide coating. A pen-like tool with a sapphire tip or ceramic scoring wafers or scribes that make a sharp cut on the column so that it may be broken cleanly in two are the best inexpensive alternatives. In any case, a fresh cut should be made and inspected just before placing the column into the inlet or detector and after sliding on the nut and ferrule.

Deactivated Fused-Silica Tubing
Gas chromatographers can use 5 or 10 m of deactivated 0.53-mm i.d. fused-silica tubing with a press-fit connector as a retention gap when necessary. Shorter pieces can act as a column-to-detector adapter when you don’t want to put the coated column end into a detector to avoid column bleed from the column end at hot internal detector temperatures.

Dental Mirror
A plastic dental mirror with a front-silvered surface makes it easy to examine the underside of an inlet fitting in the oven, or to check other inaccessible areas for loose or missing parts. The mirror can also be used to detect the flame in
a flame ionization or flame photometric detector by observing condensation of emitted water vapor on the cool mirror surface. A shiny wrench is a good substitute for the mirror in this case.

**Dental Pick**
A dental pick is very handy for removing septa from septum nuts and debris such as bits of graphite ferrule from fittings.

**Diagonal Cutters**
Diagonal cutters are used only for cutting electrical wires. They should not be used as a substitute for a tubing cutter. Don’t even think about threatening your fused-silica column with one!

**Eyedropper, Plastic**
I have a box of these that is in frequent demand. I use them to place small drops of isopropanol onto fittings for leak checks. Some types have rough volume indications more like a pipette, and I have used them to make a crude dilution of qualitative test mixtures when accuracy wasn’t required.

**Ferrules, Capillary Column**
A good assortment of graphite and graphite–Vespel capillary column ferrules is essential. Keep at least 10 of each inner diameter in the fitting sizes that match your instruments’ oven fittings. In a pinch, graphite ferrules can be squeezed into sealing on smaller columns than they were designed for. It’s always a good idea to install new ferrules with a new column; old ferrules can be reused on the same column if a seal can be made without over-tightening the fitting.

**Ferrules, Metal Tubing**
Ferrules for metal tubing are also essential. I used to prefer brass ferrules for copper and stainless-steel tubing, but nowadays I match the ferrule material to the tubing because some brass ferrules will bind to stainless steel upon tightening. Some instruments use 1/16-in. or 3/32-in. stainless tubing. This tubing is best connected using 1/8-in. graphite–Vespel reducing ferrules for 1/8-in. fittings, or 1/16-in. ferrules for the 1/16-in. tubing. Chromatographers should be aware of the potential for atmospheric oxygen contamination of the carrier gas from improperly installed supply tubing and ferrules. Even with the best filtration in place, a leak between the filters and the instrument will nullify the effect of the filters.

**Files, Needle**
An assortment of needle files can be used to pick out ferrules from fittings, remove burrs, and shape the ends of metal tubing before it is connected to a fitting. Don’t forget to clean off all traces of metal before connecting.

**Flexible Magnetic Pickup**
A flexible 2-ft magnetic pickup comes in handy when you drop a small part inside the instrument. Another similar tool has a three-jawed “claw” operated by a plunger, and it will pick up nonmagnetic items.
Flowmeter, Electronic
An electronic flowmeter is an expensive investment, but I believe that it will pay for itself many times over with improved accuracy and precision over bargain-priced bubble flowmeters. I prefer the type of electronic meter that senses flow directly and that allows the operator to select the type of gas in use, such as air, helium, or hydrogen. The option to calculate split ratios from the measured split vent and column flows is a handy feature.

Flowmeters, Bubble
If you use bubble flowmeters, keep two sizes on hand. The large size is good for measuring FID air or inlet split vent flows up to several hundred milliliters per minute. The smaller size is better for packed-column or hydrogen flame-gas flows in the 10–50 mL/min range. Don’t try to use a bubble flowmeter to measure capillary column flows below 10 mL/min. The carrier gas will diffuse out of the bubble and you will get a low reading. Measure the unretained peak time instead and calculate the flow rate from it. Note that this calculated flow rate or the rate displayed by electronic pressure control will only be as accurate as the column dimensions the operator uses. See an earlier “GC Connections” (2) for a detailed discussion.

Glass Wool Insertion and Removal Tool
This item is useful for those who must install glass wool in inlet liners, or for the hardy few who pack their own columns and use glass wool to hold in the packing. These days I find little use for it.

Inlet Liners
Inlet liners are often broken or chipped during installation or removal. They also can become contaminated with sample residue or may lose their deactivation if used for too long at high temperatures. Keep some spares on hand, both for packed inlets and for split or splitless injections. If you use deactivated liners, it is better to purchase them already deactivated than try to treat them yourself because of the chemical hazards and waste disposal problems this will create.

Inlet Liner Removal Tool
A tapered high-temperature silicone rubber tool on a metal holder does a good job of grabbing glass inlet liners and removing them without cracking or chipping the liner top. Most GC instrument manufacturers will supply specific tools and instructions for a particular inlet option.

Leak Detector, Electronic
An electronic leak detector is expensive, but it is indispensable for finding small leaks around hot fittings or inlets on which a liquid cannot be used (see “Leak-Checking Solution” below). The most sensitive type of leak detector uses a small pump to pull air from a probe through a thermal-conductivity cell. The presence of carrier gas or hydrogen changes the
thermal conductivity and causes a change in the detector’s readout compared to a reference air flow. Sensitivity for nitrogen carrier is limited. I also have a small handheld, battery-powered leak detector that has a series of light-emitting diodes which indicate the detected leak rate. This detector is great to carry around in a laboratory like mine with lots of instruments and little clear bench space.

**Leak-Checking Solution**

In my toolbox, the only acceptable leak-checking solution is a small bottle of pure isopropanol with an eyedropper. Other solutions may contain material that can leak into the gas-supply lines or columns and cause ghost peaks or other contamination.

**Magnifier**

A small magnifier is used to examine freshly-made column or tubing cuts for burrs or uneven edges.

**Manufacturer-Specific Tools**

For each GC system, there are always some specialized tools. These are used, for example, to open up a split-splitless inlet or to remove the inlet liner. Perhaps a special wrench is required for FID system flame jet replacement. Whatever the case, keep all such tools with their instrument: you will need them eventually. Some of the chromatography suppliers offer their own versions of these tools, which often are more useful than the freebie ones that come with the instruments. Several companies have toolkits for a specific popular instrument that is a must-have item for me.

**Mini Flashlight**

A flashlight is very handy for inspecting the interior of inlets and detectors for obstructions, as well as for illuminating the oven interior. I prefer the type with the bulb on a flexible gooseneck. No one yet has built a GC oven with a light that comes on when the door is opened.

**Nuts, Capillary Column**

There are several different styles of capillary column nuts that are used in the GC oven. My toolkit includes some of each type for my instruments. I never try to substitute one for another, even though the thread sizes are the same.

**Nuts, Metal Tubing**

Nuts for metal tubing are more standardized than capillary column nuts. Swaged fittings normally are used for outside tubing connections and often for internal connections as well. Keep an assortment of 1/4-in., 1/8-in., and 1/16-in. sizes on hand. Don’t try to mix nuts and fittings from different manufacturers. I’ve picked one type and tried to purge all the others from my lab, so I don’t have to peer at the small letters on each fitting to discern its type.

**Nut Drivers**

Handheld nut drivers are a useful addition, but I find that I use them more at home than in the laboratory.
Paintbrush
An artists’ paintbrush with handle is handy to clean out debris from small areas inside detectors or inlets. It can also apply leak-checking solution to fittings, although I don’t recommend this practice because of potential contamination of the gas stream with the leak-checking solution.

Paper Clips
Jumbo-size paper clips with smooth sides are convenient for blocking off inlet or detector fittings for testing purposes. Unbend the clip and attach it to the fitting with a nut and 1-mm i.d. graphite-vespel ferrule. With the column connection blocked off, you can pressure-check an inlet. A detector check can be run in this manner without column influences on noise or stability.

Pin Vise and Drills
A small pin vise and a set of drills can be used in an emergency to drill out a used ferrule or to enlarge one that is too small to fit a column. Sometimes the small drills can help to remove a ferrule that is stuck in a fitting or to remove debris from inside fittings or tubing ends.

Pliers
I keep some small needle-nose pliers, a pair of larger multigrip pliers, and one pair of locking pliers in my toolkit. The larger gripping pliers are useful for holding a straight length of 1/8- or 1/4-in. metal tubing while cutting it, although I take care not to grip the tubing anywhere near a location where a connection is to be made because the scratches from the pliers would make it impossible to get a good seal.

Press-Fit Connectors
Glass press-fit connectors make it easy to repair a broken column temporarily (until a replacement can be installed). These are available in many sizes to connect fused-silica tubing of the same or different diameter. They also connect a column with a retention gap. One manufacturer now offers a vacuum–melting device that makes near-perfect connections.

Pressure Gauge, Inlet
I have a conventional 0–60 psig pressure gauge with a syringe needle attached that I can insert into an inlet through the septum. Once in a while I need to check the inlet pressure this way, instead of relying on the instrument’s gauges or electronic pressure readouts.

PTFE Tape
PTFE tape is used sparingly on tanks and interconnecting fittings where threads form the seal. Use two layers of tape, not more, and wrap them around the threads in the direction the nut tightens so that the tape will be drawn into the fitting instead of pushed out. PTFE tape should never be used in swage-type ferrule-sealed fittings, where it will only cause a leak, nor is it used at the high-pressure supply cylinder connection. Several types of this tape are available; be sure to select the right one.
**Ruler**
A small metal ruler measures the correct column penetration depth into an inlet or detector. Don’t use a plastic ruler, because it might melt in contact with heated inlets or detectors. For convenience, make marks on the ruler that correspond to the correct inlet and detector depths. Several manufacturers offer capillary column installation gauges with the appropriate markings.

**Scissors**
A good sharp pair of scissors comes in handy for opening packages of ferrules, or for making paper stars out of waste paper that’s waiting to be recycled while watching for peaks to be eluted. Scissors are not to be used to cut fused-silica columns (but you can believe that I’ve seen someone try it).

**Screwdrivers, Phillips-Head**
I found three Phillips-head screwdrivers in my toolkit: large, medium, and small. The small one is part of a set of jeweler’s screwdrivers with rotating handles.

**Screwdrivers, Slotted-Head**
I also keep three slotted-head screwdrivers. The small one is useful for securing electrical connections to screw-type terminals. My set of jeweler’s screwdrivers in a small plastic box have a knurled body and a separately-rotating knob that make it easy to turn the shaft with one hand. They haven’t seen too much GC use, but they are good for tightening the frames of my eyeglasses.

**Seals, Inlet**
Many capillary inlets use an internal O-ring seal to isolate the incoming and exiting split flows. These seals are available in a variety of materials, including silicone, graphite, and high-temperature polymer. Worn seals will cause internal leakage and performance losses. Keep a good assortment on hand for each instrument. Some instruments use a metal seal and washer at the bottom of split–splitless inlets. For these, I prefer the deactivated seals available from at least one supplier. A seal with a Vespel seating surface for the inlet liner was recently made available, as well.

**Septa**
Spare septa are a requirement. Septa should be changed often. If you wait until retention times begin to drift upward then it’s too late—a significant leak that will compromise results already has developed. Keep both normal-temperature range septa as well as some high-temperature ones on hand. I handle my septa—and all internal inlet parts—with tweezers or cotton gloves: a little bit of finger contamination can create a significant baseline bleed level.

**Static Pad**
A static pad is a grounded, conductive plastic sheet onto which it is safe to place electronic components that must be protected from damaging electrostatic discharge. Any circuit boards removed from an instrument should be placed
on a grounded static pad, or in a static-proof bag.

Static Wrist Strap
A grounded static wrist strap prevents the technician from imparting a potentially harmful static discharge into instrumentation or components. Always wear one when working inside an instrument or removing components, and in all cases be quite sure that the instrument power has been removed while the instrument itself remains grounded.

Stopwatch, Digital
A digital stopwatch times bubbles in a bubble flowmeter, and also times an unretained peak. It’s often more convenient to use a stopwatch when setting up an instrument than to operate the chromatography data system for each test injection. Select a stopwatch with readout to 0.01 s. Some GC systems include a stopwatch function on the display that includes flow, split ratio, and linear velocity calculations. These days I just use my smart phone’s stopwatch and timer functions, and then its calculator to find flow rates or average linear velocities. Good phone applications are available with additional chromatography functions.

Syringe
I keep two syringes for setup purposes. One 10-µL syringe is for injecting methane or butane to measure the unretained peak time and ascertain that the flame is lit and carrier gas is flowing. The other is for making liquid test-mixture injections as part of a column check-out. Sample syringes are kept separately.

Syringe-Cleaning Wires
Syringe cleaning wires may be used in an emergency to clear septum particles or other debris from syringe needles. I recommend discarding stubborn contaminated syringes; take steps to keep the syringe clean instead.

Test Mixtures
Column and detector test mixtures verify column performance and detector sensitivity. Keep a fresh vial of each type on hand. Column test mixtures are available for polar and nonpolar capillary columns, and there are test mixtures for each detector type. Some manufacturers provide a detector test mix that combines components for testing several different detectors. After they have been opened, test mixtures can be kept for a while in septum-sealed vials. Their lifetime is limited because of gradual evaporation. If you keep test mix in a vial, remove the vial cap rather than puncturing the septum when withdrawing liquid for injection. Some laboratories find it more convenient to keep dilute test mixtures on hand because these are more easily disposed of than the concentrated mixes. Many laboratories have their own qualification and validation standards, of course, but the manufacturer’s mixtures allow easy comparison to the factory test results.
Tube Bender
I use this simple tool to make controlled bends of copper or stainless steel tubing for connecting the supply tanks to the filters and then to the back of the instrument. Tube benders come in sizes to fit standard tubing diameters.

Tube Reaming and Deburring Tools
These tools are used to remove burrs and irregularities from metal tubing after cutting. They are available for the standard tubing diameters, and I highly recommend using them to ensure leak-free connections.

Tubing, Plastic and Rubber
I keep several pieces of black and clear silicone rubber tubing on hand for connecting my flowmeter to column ends, split vents, and other flow sources. The narrower pieces of tubing fit inside the wider ones so that I can adapt the flowmeter fitting to a variety of connections. Of course, I never use plastic or rubber tubing for any gas at elevated pressure or for permanent supply or internal connections.

Tweezers and Hemostats
A pair of tweezers can hold small nuts or ferrules without risking contamination with skin oils or a burn from hot items. Some tweezers have a convenient locking feature that frees one hand for other tasks, as will a spring-loaded hemostat. Rubber tips help hold fragile capillary columns or inlet liners.

Vial Crimper
Vial crimpers attach aluminum crimp-top seals to autosampler vials. Several crimp-top sizes are commonly used for GC: 8 mm for 0.8-mL vials, 11 mm for 1.5- or 2.0-mL vials, and 20 mm for 5-mL and 20-mL vials. Hand crimpers are the least expensive, and some are available with interchangeable jaws that accommodate different vial sizes. Automated benchtop crimpers are less mobile, but the jaws can be interchanged quickly and they are best for laboratories with high sample throughput.

Vial Decapper
Vial decappers perform the opposite function of a crimper: They remove crimp-top seals from vials. Decappers come in the same sizes as the crimpers and resemble a pair of pliers. Some caution is required when using these so as not to break the neck of the vial. After the caps are removed, the contents may be properly disposed. Some laboratories reuse sample vials, but I recommend a fresh vial for each sample if at all possible.

Vial, Autosampler
I keep a spare autosampler vial to check for carrier gas flow during column installation. Fill the vial halfway with distilled water and then insert the column outlet after connecting to the inlet and turning on carrier gas pressure. The presence of bubbles shows positive carrier gas flow.
**Wipes, Laboratory**
I wet these with some isopropanol and then clean any debris or oil off the ends of capillary columns before inserting them into inlets or detectors. They also are handy for tipping a drop of test mix off a syringe needle, if disposed of properly. Paper towels don’t work as well: They may leave fibers behind, and they could deposit a chemical residue.

**Wire Brushes**
Wire brushes can dislodge particles and debris from detector parts and some sealing surfaces. Be careful not to score polished metal surfaces, or damage ceramics. It is better to replace a severely dirty FID flame jet or collector than to clean it forcibly.

**Wrenches, Hexagonal and Star**
A full set of inch and metric hexagonal wrenches comes in handy when some minor disassembly is required.

**Wrenches, Open-Ended**
I have an assortment of open-ended wrenches in inch sizes as well as a metric set. I keep two or three with the following sizes: 1/4, 5/16, 3/8, 7/16, 1/2, and 9/16 in., as well as 11/16, 3/4, and 1 in., although these latter sizes are used only rarely. I apply two wrenches at once to prevent counter-rotation while tightening or loosening fittings.

**Wrenches, Adjustable**
I have one large 18-in.-long adjustable wrench that looks like it belongs in an automotive garage. This is used exclusively for attaching or removing pressure regulators on gas tanks. I also have a smaller 6-in. long adjustable wrench that I use occasionally if someone else has walked off with the exact open-ended wrench size I need.

**Conclusion**
Chromatographers, like all craftspeople, use a variety of tools to practice their craft. In a pinch, tools that are somewhat inappropriate can be used to make do, but the rapidity and ease with which the right tool gets the job done make it well worth the expense of obtaining what’s needed.

**References**

John V. Hinshaw is a Senior Scientist at Serveron Corporation in Beaverton, Oregon, and a member of LCGC’s editorial advisory board.

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Proven Consumables
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- GC derivatization reagents
- Gas flowmeter & leak detector

GC Columns and Consumables
In terms of selecting an appropriate stationary phase for gas chromatography (GC), there are four primary analyte or stationary-phase interactions that need to be considered.

Dispersive interactions (<<1 kJ/mol) are lower energy (van der Waals) forces between nonpolar moieties of the analyte molecule, that is, C-H bonds and so on. These will be in play when using any silica-based stationary phase because the majority of the polymeric backbone (polydimethylsiloxane [PDMS]) of a silica-based stationary phase is nonpolar in nature.

Dipole–dipole and dipole–induced dipole interactions (3 and 1 kJ/mol, respectively) are in play whenever unsaturated, aromatic, or more-polar functional groups (that is, C-Cl or C-N bonds) are present in the stationary phase or analyte molecule. Stationary phases containing phenyl, cyano, or trifluoro functional groups are more polar than PDMS, and the more of these functional groups there are, the greater their influence is on the separation. The increase in retention of aromatic compounds and the relative decrease in retention of aliphatic analytes when moving from a 5% phenylmethyl PDMS phase to a 50% phenylmethyl PDMS phase exemplifies this.
Hydrogen bonding interactions (19 kJ/mol) are the strongest intermolecular forces in capillary GC and occur whenever the stationary phase contains cyano, trifluoro, or (especially) hydroxyl functional groups. This type of force is in play when analyzing alcohols using a polyethylene glycol or “wax” type phase.

Pragmatic phase-selection rules can be summarized as follows:

- Use the principles of “like dissolves like” wherever possible and match the polarity of the analyte to the polarity of the stationary phase.
- Remember that really there are only five “chemistries” we need to consider (see Figure 1). To increase retention or alter selectivity based on a particular interaction, increase the amount of the functional group within the phase (that is, move from a 14% to a 35% cyanopropyl phase).
- Use the least-polar phase possible because more-polar phases bleed more (it’s inherent in the chemistry).
- A 5% phenyl column can be used to screen unknown samples — analyte retention and selectivity can then be assessed and a more appropriate phase chosen if necessary.
- A 5% phenyl, 50% phenyl, 14% cyanopropyl, and a wax (polyethylene glycol [PEG]) column cover the widest range of possible interactions.

Figure 1: The five most common GC column stationary-phase chemistries.
(stationary-phase polarities) in the fewest number of columns. How does one select the physical column dimensions of length ($L$), internal diameter, and film thickness ($d_f$)?

Column length affects the separation efficiency and therefore the resolution. Doubling column length doubles efficiency (number of theoretical plates [$N$]), doubles analysis time in isothermal separations (1.5–1.75× increase if using gradient temperature programming), doubles column cost, and increases resolution by a factor of 1.4. Increasing column length is the worst way to improve the resolution of a separation; however, when you have a sample with many components (hundreds), you sometimes need a long column. Select column length according to the number of species that need to be separated in the sample. For two components, use a 10-m column, and for hundreds of components, use a 60-m or 120-m column.

Column internal diameter affects retention and efficiency. Halve the column internal diameter, double the efficiency, and increase resolution by a factor of 1.4. This will double retention time only for isothermal separations and only if the film thickness in not altered. The phase ratio ($\beta$) is equal to the column radius (mm) divided by $2 \times$ the film thickness (µm). Keep the phase ratio constant between columns and the retention time will be approximately constant. Use $\beta < 100$ for highly volatile analytes and $\beta > 400$ for high-molecular-weight analytes or for trace analysis. Use smaller internal diameter columns when the separation is dependent on the stationary phase selectivity (analytes chemically very similar) or when multiple components need to be separated in shorter timeframes. Note that the column capacity will decrease as the column internal diameter is reduced.

Film thickness affects retention of analyte species, interaction with the silica tubing, phase bleed, and column capacity. Doubling the film thickness doubles retention time for isothermal analysis and increases retention by a factor of around 1.5 for temperature-programmed analysis. Doubling film thickness increases elution temperature by around 20 °C. Use thin films (0.1–0.25 µm) for trace analysis or when analytes are relatively involatile. Use thicker films (1–5 µm) when dealing with volatile analytes, analytes at high concentration, or when peak shape is poor. Note that increasing film thickness may compromise resolution for later-eluted analytes (retention factor > 5) and that phase bleed and column capacity increase with increasing film thickness.

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