Connections

GC Troubleshooting — A Troubleshooter's Tool Kit

This month in "GC Connections" John Hinshaw examines the tools and accessories that gas chromatographers use in their laboratories.

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Every profession has its specialized tools. The tools 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 also are found in plumbers', carpenters', and homeowners' tool kits. Wrenches, screwdrivers, pliers, and tubing cutters are some easily recognized examples. Other items such as dental instruments or correction fluid are familiar, but their use in laboratory environments might not be obvious immediately. Still others, such as column flowmeters and specialized fused-silica–column cutters, aren't found outside laboratories at all.

When I looked through my laboratory toolbox recently, I found some new items that hadn't been in it when I wrote the first column about tools, back when "GC Connections" was called "GC Troubleshooting" (1). I also found other items that were included in the original list but hadn't been used very much since. This month's column is a list of today's gas chromatography (GC) tools and accessories, with some information about their use and significance. One or more specialty manufacturers offer many of the chromatography-specific items: I've tried to keep the descriptions as generic as possible, but mention of specific tools sometimes was unavoidable. This mention doesn't imply an endorsement on the part of myself or *LCGC;* individual chromatographers must determine whether the tools are suitable for their individual applications.

Butane lighter: A lighter is a convenient source of 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 greater than 50. Columns at low temperatures or with lower phase ratios (thick stationary films) can separate the traces of ethane and propane present in the butane fuel, as can many of the gas–solid phases such as porous polymers or molecular sieves. 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. 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 an unretained substance.

Cable, shielded three-wire: So-called *recorder cable* is indispensable for new instrument installations because cables are rarely available, and many instruments are shipped without them. I have found several cables terminated with both bare wires and spade lugs, as well as some with instrument-specific connectors on the ends.

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 fluid for data-integrity reasons. If the column-oven temperature remains below approximately 200 °C, a septum can be slid onto the column before the nut and ferrule to act as a positioning aid. A positioning gauge (see **Ruler**) is a good alternative.

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 an 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. Sapphire or ceramic scoring wafers or scribes that make a sharp cut on the column so that it can be broken cleanly in two are the best inexpensive alternatives. In any

case, a fresh cut should be made just before placing a column into an inlet or detector, after sliding on the nut and ferrule.

Cutters, tubing: A large plumber's tubing cutter for ¼-in. metal tubing and a smaller one for ⅛-in. tubing are used extensively during instrument installation and gas-supply setup. Keep a supply of new cutter blades. These disks wear out rapidly, and a dull blade will distort the tubing diameter and make it difficult to slide on the back and front ferrules. Power cutters with high-speed rotary blades also are available. These cutters are mandatory for cutting 1/16-in. tubing for low dead-volume gas valving applications. After cutting and squaring the tubing, be sure to clean the ends with a solvent such as isopropanol so that loose particles can't get into gas-switching valves or interfere with the passage of peaks.

Deactivated fused-silica tubing: Gas chromatographers can use 5–10 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 column-to-detector adapters 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 also can be used to detect the flame in flame ionization or flame photometric detectors by observing condensation of emitted water vapor on the cool mirror surface. A shiny wrench is a good substitute for a mirror in this situation.

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.

Ferrules, capillary column: A good assortment of graphite and graphite–Vespel capillary column ferrules is essential. Keep at least 10 of each inner diameter size in ⅛-in. and 1/16-in. fitting sizes that match your instruments' oven fittings. In a pinch, graphite ferrules can be squeezed into sealing on columns smaller than they were designed to fit. 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 overtightening the fitting.

Ferrules, metal tubing: Ferrules for metal tubing also are essential. I prefer brass ferrules for copper and stainless steel tubing in ¼-in. and ⅛-in. diameters. Some instruments use 1/16-in. or 3/32-in. stainless tubing. This tubing is best connected using ⅛-in. graphite–Vespel reducing ferrules for ⅛-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.

No one yet has built a GC oven with a light that turns on when the door is opened.

Files, needle: An assortment of needle files can be used to pick out ferrules from fittings and to remove burrs and shape the ends of metal tubing before it is connected to a fitting.

Flexible magnetic pickup tool: A flexible 2-ft magnetic pickup tool comes in handy when you drop a small part inside an instrument. A 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 compared with the more conventional bubble flowmeters. I prefer the type of electronic flowmeter that senses flow directly and that allows operators 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 another handy feature.

Flowmeters, bubble: If you use bubble flowmeters, keep two sizes on hand. The large size is good for measuring flame ionization detector air or inlet split vent flows at rates ranging from 100 mL/min to 1000 mL/min. 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 less than 10 mL/min. The carrier gas will diffuse out of the bubble, and you will obtain a low reading. Measure the unretained peak time, instead, and calculate the flow rate from it.

Inlet liners: Inlet liners often are broken or chipped during installation or removal. They also can become contaminated with sample residue or can lose their deactivation if used for too long at high temperatures. Keep some spare liners 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.

Inlet-liner removal tool: This 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.

Leak-checking solution: In my toolbox, the only acceptable leak-checking solution is a small bottle of pure isopropanol with an eye dropper. Other solutions can contain materials that can leak into the GC lines and cause ghost peaks or other contamination.

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. Sensitivity for nitrogen carrier is limited, because the leak detector uses air to establish the zero point. I also have a small, handheld, battery-powered leak detector that has a series of light-emitting diodes to indicate the detected leak rate. This detector is great for carrying in a laboratory with many instruments.

Magnifier: A small magnifier can be used to examine freshly made column or tubing cuts for burrs or uneven edges. I have both 10× and 5× varieties.

Manufacturer-specific tools: Each gas chromatograph always has some specialized tools. These tools can be used, for example, to open up a split–splitless inlet or to remove an inlet liner. Perhaps a special wrench is required for flame ionization detector flame jet replacement. Whatever the case, keep all of these tools with their associated 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. One company manufactures a tool kit for a specific popular instrument that is a must-have item for me.

Miniature 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 turns on when the door is opened.

Nuts, capillary column: Several different styles of capillary column nuts are used in GC ovens. My tool kit 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 ¼-in., ⅛-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 laboratory, so I don't have to check each fitting to see its type.

Paintbrush: An artists' paintbrush with a handle is handy to clean debris from small areas inside detectors or inlets. It also can apply leak-checking solution to fittings, although I don't recommended 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 and 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. You can perform a detector check 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 remove a ferrule that is stuck in a fitting.

Press-fit connectors: Glass press-fit connectors make it easy to repair a broken column temporarily (until a replacement can be installed). These connectors are available in many sizes to connect fused-silica tubing of the same or different diameters. They also can connect a column with a retention gap.

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: Polytetrafluoroethylene (PTFE) tape can be used sparingly on tanks and interconnecting fittings where the threads form the seal. Use two layers of tape, no 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 being pushed out. PTFE tape is not used in swage-type fittings in which it would cause a leak, nor is it used at the high-pressure supply cylinder connection.

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.

One manufacturer offers a capillary column installation gauge that preseats the ferrule in position.

Scissors: A good, sharp pair of scissors comes in handy for opening packages of ferrules or for making paper stars out of all the antiquated chart paper while you are waiting for peaks to be eluted.

Screwdrivers, Phillips-head: I found three Phillips-head screwdrivers: 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. I also have a set of jeweler's screwdrivers in a small plastic box. These 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 my eyeglass frames.

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 instruments, I prefer the deactivated seals available from at least one supplier. A seal with a Vespel seating surface for the inlet liner recently appeared on the market, as well.

Septa: Spare septa are a requirement. Septa should be changed every 50–100 injections or as soon as retention times begin to drift upward. Keep both normal-temperature-range septa and 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 oil 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 technicians from imparting a potentially harmful static discharge into instrumentation or components. Always wear one when working inside an instrument or removing components.

Stopwatch, digital: A digital stopwatch times the 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 gas chromatographs include a stopwatch function on the display that includes flow, split ratio, and linear velocity calculations.

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

Syringe-cleaning wires: Syringe cleaning wires can 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 of test mixture on hand. Column test mixtures are available for polar and nonpolar capillary columns and for each detector type. Some manufacturers provide a detector test mix that combines components for testing several different detectors. After they are opened, test mixtures can be kept for a while in septum-sealed vials. Their life is limited due to gradual evaporation. If you keep a test mixture in a vial, remove the vial cap rather than puncture the septum when withdrawing liquid for injection. Some laboratories find it more convenient to keep very dilute test mixtures that are more easily disposed of than more-concentrated mixtures.

A good, sharp pair of scissors comes in handy for opening packages of ferrules or for making paper stars out of all the antiquated chart paper.

Tubing 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 backs of the instruments.

Tubing, plastic and rubber: I keep several pieces of black and 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 various connections. Of course, I never use plastic or rubber tubing for any gas at elevated pressure nor for permanent supply or internal connections.

Tweezers: 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. 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 — 8 mm for 0.8-mL vials, 11 mm for 1.5- or 2.0-mL vials, and 20 mm for 5- and 20-mL vials — commonly are used for GC applications. Hand crimpers are the least expensive versions. The crimpers with interchangeable jaws that accommodate different sizes are a little more expensive. The expensive bench-top crimpers are less mobile, but the jaws can be interchanged quickly, and they are best for high-sample-throughput laboratories.

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. They resemble a pair of pliers. Some caution is required in their use to avoid breaking the neck of the vial. After caps are removed, the contents can be disposed of properly, and the vial can be cleaned and reused if appropriate.

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 flame ionization detector flame jet or collector than to clean it forcibly.

Wrenches, open-ended: I have an assortment of open-ended wrenches in inch and metric sizes. I keep two or three wrenches with the following sizes: 1/4, 5/16, 3/8, 7/16, 1/2, and 9/16 in. I apply two wrenches simultaneously to prevent counter-rotation while tightening or loosening fittings.

Wrenches, adjustable: I have one large 1-ft-long adjustable wrench that looks like it belongs in an automotive garage. I use it 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 has walked off with the exact open-ended wrench size I need.

Reference

(1) J.V. Hinshaw, *LCGC* **11**(2), 96–102 (1993).

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