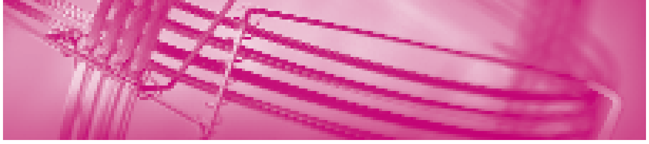

Gas Cylinder Safety, Part I — Hazards and Precautions

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Many gas chromatographers are not fully aware of safe practices for handling high-pressure gas cylinders. Operators should be trained to properly transport, install, connect and maintain their gas supplies, as well as to deal with emergencies. In the first of a two-part series, this month's "GC Connections" examines the principal hazards and safety issues surrounding the compressed gas cylinder. The second instalment will present procedures for routine cylinder use.

A nightmare: It's Monday morning and a co-worker has found that one of the helium cylinders in the laboratory had emptied over the weekend. I watch as he reaches over the other gas cylinders, applies the big tank wrench and accompanied by a loud hissing sound, detaches the regulator fitting from the tank. Letting the regulator hang by its plastic connecting tubing, he moves the hydrogen and air cylinders into the corridor between the laboratory benches, tilts the empty cylinder on its bottom edge and rolls it into position near the door. He leaves the laboratory and returns in a moment pushing a furniture dolly in front of him. With a grunt, he tilts the cylinder sideways onto the dolly and pushes it down the hallway. Ten minutes later, he returns with a new cylinder on the dolly, which rolls away and bangs against the laboratory bench as he lifts the tank up to a vertical position. He removes the cylinder cap, holds it in one hand and uses his other hand to examine the cap that had come a couple of weeks ago with the empty tank. He puts both caps down with a shrug and grasps the neck of the new tank's exposed valve. With a grimace he ducks down slightly, cracks open the valve, and is rewarded with a 110 dB roar as the escaping gas expresses its new freedom. Satisfied with the demonstration, he rolls the tank into position and reattaches the regulator. Then he begins to return the other tanks to their original positions. Someone near the door calls his name loudly and as he spins around to see who it is, his belt buckle catches one of the dangling gas lines. In dreamlike slow motion, the hydrogen tank begins to head for a horizontal position as its valve and regulator glance off the bench top on the way down. A bright orange-yellow light fills my eyes … and I wake up with the morning sun streaming onto my face.

Certainly, no one would take all of the wrong actions that my dreamworld co-worker did, but how many of us have done just one of them? I've witnessed them all, and I'm guilty of a few myself from time to time, especially in exceptional situations such as setting up a demonstration in a conference room. I sincerely hope that everyone working in a laboratory treats flammable solvents and toxic chemicals with well-deserved respect and understands the short- and long-term risks involved with handling hazardous materials. So what leads some of us to fall short of giving compressed gas cylinders the respect they deserve? In terms of stored potential kinetic energy, they are bombs waiting to explode; in terms of suffocation potential or flammability, they can be as great a fire hazard and potentially as toxic as many solvents and solids.

Let's take a look at the hazards gas cylinders present and some procedures and practices that can maximize safety for those who work with them.

Cylinder Hazards

Gas cylinders present several obvious and some less-familiar hazards, including sudden decompression that can propel a cylinder remarkably quickly across a laboratory and displace breathing air; risk of explosion or reaction; possible acute toxicity; heavy-object hazards; and personal injury from high-pressure gas streams or cryogenic liquids. The Occupational Safety and Health Administration (OSHA) regulations *29 CFR Parts 1910.101–1910.105* provide specific guidelines for the use of compressed gases in the workplace. Gas chromatographers do not normally use some of the common hazardous gases such as acetylene, oxygen, nitrous oxide and propane in pure form, but they may encounter them in other laboratories and should be aware of the extra dangers that chemically reactive, fuel, or oxidizer gases pose. Table 1 lists hazard classes for commonly used gas chromatography (GC) gases.

Several commonly accepted first aid procedures address exposure to these hazards. However, I am no expert in this area. I strongly recommend that all personnel who use compressed gases be trained in basic first aid and that a few receive additional cardiopulmonary resuscitation (CPR) and other advanced training.

Rocket ship: The first thought that comes to my mind when discussing gas cylinders is their rocket potential. A 1-A cylinder of helium contains 8.3 m^3 (293 ft^3) of room-pressure gas that's compressed into a space of less than 0.5 m^3 (2.0 ft^3) at a pressure of 18.1 MPa (2640 psi). The European L-size cylinders contain slightly more compressed gas. These cylinders weigh approximately 91 kg (200 lb) when empty and the weight of the helium within a fully pressurized cylinder is approximately 1.4 kg (3 lb). When the gas pressure is released rapidly through an opening the size of the valve stem, the cylinder — if it shoots in a straight line — can reach velocities of close to 30 m/s, 108 km/h or 66 mph. A 91 kg metal cylinder hurtling at high velocity can do tremendous damage almost instantaneously and a person can do nothing to stop it after a decompression incident starts. See the sidebar "How Fast Will a Cylinder Fly?" for the calculations that produced this velocity figure.

The thought of a heavy cylinder careening through the laboratory walls gets the attention of most chromatographers. This type of accident, however, is easy to avoid by always restraining cylinders with appropriate chains or brackets, transporting them in cylinder carts and keeping them capped at all times unless they are actually in use with a regulator or manifold attached. Any cylinder found to be damaged or that has a stuck valve should be returned immediately to its supplier. If the damage is to the cylinder body, the supplier should be notified and asked to remove it. Never try to vent a damaged cylinder.

Atmospheric displacement: Another problem can occur when the contents of any large gas cylinder — other than an air cylinder — vent rapidly, even if the cylinder is restrained. The sudden release of more than 8 m^3 of unbreathable gas in the laboratory will reduce the level of oxygen in the air dramatically and present a real suffocation hazard. Liquefied gases expand as much as 1000-fold in volume when vaporized and can present a much greater hazard. Large liquid nitrogen Dewars contain enough nitrogen gas to make a room incapable of sustaining life if the gas is released rapidly. Carbon dioxide can cause immediate unconsciousness followed by death when breathed in any significant concentration. It is much denser than air and will settle in low, unventilated areas. Liquid carbon dioxide tanks, such as the ones used for GC cooling, can release especially large quantities of gas during a tank rupture.

If an event such as this happens, leave the area immediately, prevent others from entering the area and seek the assistance of personnel trained in the use of a self-contained breathing apparatus. Make sure the area has been well ventilated before returning. Don't try to reenter a hazardous area without the proper breathing equipment to assist someone else. Many unnecessary tragedies have occurred because of misguided rescue attempts.

Explosion and fire hazards: If a hydrogen cylinder vents into a laboratory in an uncontrolled manner, even if the leak is through the pressure-release disk on the cylinder or regulator, leave the area immediately, close the doors, pull the fire alarm to evacuate the building, and call emergency services. Don't try to extinguish flame detectors or shut down anything in the laboratory — just get out quickly. Hydrogen has a flammable concentration in air of 4% so a venting cylinder can easily create an explosive concentration in moments. In its favour, hydrogen rapidly diffuses in air so the flows typically encountered with flame detectors or carrier gases present no significant hazard under normal conditions.

The same evacuation procedure is required with other flammable gases such as propane and acetylene or reactive gases and oxidizers such as oxygen and nitrous oxide. Breathing air contains approximately 20% oxygen, but high oxidizer concentrations will accelerate combustion dangerously and can cause serious burn injuries. Remember that clothing, paper, paint and plastic can all burn rapidly in the presence of high oxidizer concentrations.

If a gas fire starts and the gas leak cannot be stopped safely and positively, don't try to extinguish the flame. Unburned gas can accumulate and explode if an ignition source is present. Hydrogen particularly presents a special hazard because it burns in excess air with an invisible flame. Never try to investigate a possible hydrogen fire by approaching the suspected flame area; leave it to the professionals. Although the combustion by-products of hydrogen are non-toxic, the fire can burn other nearby items such as

Table 1: Hazard classes for commonly used GC gases and other gases found in the laboratory.

	Decompression	Flammability	Asphyxiation	Toxicity	Cryohazard
Acetylene	U	U	U		
Air	U				
Argon	U		U		U*
Carbon dioxide	U		U	U	U*
Chemical reagents (reactive compressed gases)	U	U	U	U	
Helium	U		U		U*
Hydrogen	U	U	U		U*
Nitrogen	U		U		U*
Oxygen	U	U†			U*
Propane	U	U	U		

* Liquefied gas
† Accelerates combustion

plastics, which can produce toxic combustion by-products.

High-pressure gas cylinders can rupture explosively when heated in a fire. All cylinders include a thermal fuse that is supposed to melt and release the cylinder contents in a semicontrolled manner before the internal pressure reaches a safe upper limit. However, if the cylinder has been mechanically stressed by a fall or the impact of another cylinder, it can burst before the pressure relief valve can act. A chain-reaction effect sometimes occurs in large fires in areas where many cylinders are stored.

Toxicity: GC gases aren't generally toxic. That is, after a victim has been removed from an accident area and has received first aid, the immediate effects of gas exposure such as dizziness and difficulty breathing will diminish rapidly. Sample or reaction gases, alternatively, can present a real toxic health hazard and a significant disposal problem. If even a small leak of a toxic gas is detected, leave the area and call trained personnel to move the leaking cylinder to a safe place. Each type of gas has an associated material safety data sheet, which must be sent in advance to the purchaser, who must then keep the information on file for access by any employee. Material safety data sheets contain extensive information about the use, storage and disposal of chemicals, including compressed gases; their toxicity; and any other relevant information. Refer to the appropriate material safety data sheet when you have any questions about a particular material.

How fast will a cylinder fly?

Let's assume that helium is allowed to vent unobstructed through a 1.1 cm (0.5 in.) orifice, such as the cylinder valve stem, during a 10 s interval. That's just my guess at a time frame that seems reasonable. The thrust or force exerted on the cylinder at any moment will be the sum of two terms: the mass flow of the helium times its exit velocity through the orifice and the pressure differential across the orifice times its area, as delineated in Equation 1:

$$F = qV_e + A_e\left(p_e - p_a\right) \qquad [1]$$

where q is the rate of helium mass flow, V_e is the exit velocity through the orifice, A_e is the orifice cross-sectional area, and p_e and p_a are the cylinder and ambient pressures, respectively.

The helium must expand through the orifice, which has an area of 0.000095 m^2, into an 8.3 m^3 volume in 10 s, which produces an average exit velocity during the release of 87 m/s. That's approximately 314 km/h, 200 mph or 25% of the speed of sound, and these numbers accentuate the hazards of rapid decompression. The exit velocity will be higher at first and then slow as the tank pressure decreases. This reaction mass of the helium will impart an average force of approximately 12 kg m/s^2. Acting for 10 s against the mass of the cylinder — we'll ignore the loss of the helium's mass — this average force will impart a velocity change of approximately 4.8 km/h or 3 mph. That number is not very impressive, but it seems right for a relatively small mass of helium acting against a heavy cylinder.

The rocket effect primarily comes from the second term of Equation 1, which involves the high-pressure drop from the cylinder to the atmosphere. At the first instant of decompression from a full cylinder at 18.1 MPa, a force of 1710 kg m/s^2 will be exerted by the pressurized gas across the orifice. This force is so much larger than the first term that we can ignore the helium reaction mass effect, as Equation 2 shows below. As the remaining gas pressure drops, the force will also decrease and reach zero after 10 s, for all practical purposes. Recalling that $F = ma$ (force equals mass times acceleration) and then integrating the decreasing acceleration across time, Equation 2 describes the situation for an exponential decay in pressure:

$$v = \frac{A_e\left(p_e - p_a\right)}{m} \times \int_0^{\infty} e^{-kt}dt = \frac{A_e\left(p_e - p_a\right)}{mk} \qquad [2]$$

where v is the cylinder velocity after the gas has escaped, k is the pressure decay constant, and t is the time interval. A pressure decay rate of 50%/s, where k is $1 - 1/e$ or 0.632, reduces the pressure to less than 0.2% after 10 s. With a 91 kg cylinder mass, the terminal velocity is approximately 30 m/s, 108 km/h or 66 mph. Even if the pressure drop decreased more rapidly and approached zero after 5 s, the velocity would still be as high as 19 m/s, 68 km/h or 42 mph.

Many years ago, I saw lecture bottles of methyl bromide, cyanogens, hydrogen fluoride, carbon monoxide and various highly reactive silanes — not all in the same laboratory — carelessly stored on shelves above floor level with unprotected valves. No analytical or chemical laboratory can justify operation under such hazardous conditions. Improperly stored or deployed toxic gas cylinders have no place in anyone's workplace. If any are found, it's good procedure to evacuate the area and call a hazardous materials team to remove the danger. In any case, never try to move or dispose of hazardous or unknown chemicals in gaseous, liquid or solid form by yourself — it's not worth the risk.

Heavy lifting: No one should attempt to lift a cylinder that weighs more than approximately 12 kg (26 lb). Heavy cylinders belong on the floor and they should be restrained to a bench or a wall. Always use a cylinder cart to move cylinders, even from one part of a laboratory to another. The practice of rolling a cylinder on its bottom edge, although prevalent, risks injuries to feet and toes — and risks a cylinder becoming unbalanced and falling. Never place a cylinder on its side and roll it; the sidewalls are the thinnest parts of a cylinder and aren't designed to support any weight. You might create a dangerously weak cylinder that could explode the next time it's filled with gas.

Liquid carbon dioxide cylinders, which are used for cryogenically cooling GC components, weigh much more when full because of liquid carbon dioxide's density, and they can be deceptively heavy. Always pay special attention to these cylinders. In all instances, it's good practice to wear protective eyewear, shoes, gloves and clothing when manipulating large gas cylinders.

Cryocooling: Cryogenic liquefied gases such as liquid nitrogen or carbon dioxide present additional hazards in the laboratory. Carbon dioxide, a liquid when stored under pressure at room temperature, cools to subzero temperatures when decompressed because of both expansive and evaporative cooling. Liquid nitrogen is stored under low positive pressure in a special Dewar tank at −195 °C. Both liquefied gases can cause immediate frost burns on exposed skin. Liquid nitrogen also presents a cryogenic freezing hazard that embrittles almost any object it contacts in bulk, including fingers. Connecting tubing that conducts cryogenic liquids also presents a freezing hazard — the tubing should always be insulated or shielded to prevent accidental contact. Again, appropriate protective wear such as thermal gloves, eyewear and skin-covering clothing helps prevent accidents.

High pressure: The co-worker in my nightmare liked to crack open the high-pressure valve without a regulator attached. I suppose his idea was to blow out any dust particles and to see if the tank was pressurized, but this behaviour is never a good idea. The force exerted by gas decompressing from high pressures is tremendous. If he happened to have part of his hand or arm in front of the cylinder fitting, he could have suffered a serious abrasion, deep cut or worse. A much better way to clear the dust is to spray the area with clean, dry compressed air from a good air source or a small can. Never spray a halocarbon-based material onto the cylinder fitting — the gas can get into the lines and cause problems with electron-capture and mass spectrometry detectors.

Conclusion

I've addressed many of the hazards associated with compressed and liquefied gases in this month's "GC Connections." The four most important considerations when dealing with compressed gas cylinders are proper physical restraint, personal protection, knowledge of potential hazards and appropriate emergency procedures. After a cylinder is in place in a laboratory, the next step is to hook it up and put it in service. In the next article, I'll present some good procedures to follow when installing, using and replacing gas cylinders and pressure regulators.

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For an ongoing discussion of GC issues with John Hinshaw and other chromatographers, visit the Chromatography Forum discussion group at http://www.chromforum.com

Gas Cylinder Safety, Part II — Set-up and Use

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In last month's "GC Connections," John Hinshaw discussed some fundamental issues concerning gas cylinder hazards and presented common-sense precautions that all laboratories should implement. In this month's instalment, he examines procedures for the safe set-up, use and disposal of compressed gas cylinders in chromatography laboratories.

Compressed gases are the unavoidable companions to gas chromatography (GC). GC carrier, detector and make-up gases must all be supplied to instruments at pressures that are greater than the highest anticipated delivery pressure to the columns and other gas-dependent devices. To attain large stored-gas volumes, gas suppliers sell compressed gases for GC use in a variety of high-pressure cylinders. Pressure regulators act as the intermediaries between cylinder pressures and pressure levels that are compatible with instrumentation. With internal pressures that can exceed 18 mPa (2600 psig), these cylinders present a rapid decompression hazard, as well as flammability, asphyxiation, toxicity and cryohazards. Laboratories that use compressed gas cylinders should implement proper storage, transportation, handling, installation and disposal procedures, and appropriate emergency plans and then train all laboratory personnel and any others who may encounter compressed gas cylinders.

The first step towards the safe use of gas cylinders in laboratories is their restraint in place. The selection and installation of appropriate pressure regulators, fittings and tubing between the cylinders, the gas supply filters and the GC gas inlets are all critical for successful gas safety as well as for performance. After they are operational, gas cylinders require occasional monitoring of the remaining gas pressure until it drops below a useful level. Then, it's time to disconnect the pressure regulator, return the used tank and install a full one. I'll examine these issues in detail in this month's "GC Connections" column. (In last month's column, I discussed fundamental issues of gas cylinder hazards and described precautions that every laboratory should implement.)[1]

Hooking Up

The most important steps towards safe and effective use of compressed gas cylinders are installing and setting up the cylinder, the associated regulator and the connecting tubing. Gas filters are a separate consideration and one that is beyond the space limits of this "GC Connections" instalment.

Cylinder transport and restraint: Most cylinder accidents occur during transport or are caused by improper restraint. By taking a few simple precautions and following some guidelines, gas chromatographers can prevent all accidents of this type. Although proper cylinder handling does take a few minutes longer than other approaches, the time spent is a necessary cost for attaining the safest possible laboratory environment.

Cylinder transport: Never leave a cylinder standing unrestrained, even temporarily while it's being replaced or installed. A dual-cylinder cart is handy when exchanging cylinders. Bring the new cylinder on the cart to its location, secure the used cylinder on the cart in the free position, and only then remove the new cylinder and install it. Of course, no one should try to roll a cylinder on its bottom edge any significant distance across the floor. Although it's necessary to do this to move a cylinder between its restraint and a cart, that's the limit. It's also a good idea to wear heavy gloves when moving cylinders. They will keep your hands clean and protect them from minor injuries such as getting fingers stuck between two cylinders.

Restraint: Each gas cylinder must be restrained properly to a fixed object such as a permanently installed bench or the wall of the laboratory. Gas suppliers and other GC supply companies offer a variety of cylinder restraints for different situations. For a temporary set-up, cylinder bench clamps can be installed and removed easily; they can accommodate one or more cylinders. Wall-mounted restraints with chains or straps can be used for more permanent installations. Larger cages and racks are also available. Nevertheless, the objective is to restrain the cylinders so that they cannot be tipped over or damaged while in place. The restraining straps or chain must be adjusted tightly around the cylinders at the proper height. If they are too low, the cylinders could tip over; if they are too high, the cylinders might slip under the restraints.

Cylinder caps: Cylinder caps are designed to protect the cylinder valve and prevent sudden decompression in the event that a cylinder falls or is hit by something. The cylinder cap should remain in place on its cylinder at all times, except when the cylinder is attached to a regulator and is in service. Always attach the cylinder cap before freeing cylinders from their restraints. Store the caps in plain sight immediately next to the cylinders as a reminder to use them.

Multiple cylinders: If multiple cylinders are restrained by one device, workers must exercise extreme care to avoid losing control of any cylinder when installing or removing one. It's best to shut off all the cylinders' high-pressure valves before loosening the restraints. If a cylinder attached to a regulator must be moved out of the way, the regulator should be detached from the cylinder (see below for a discussion of this procedure) and the cylinder cap must be installed before moving it.

Regulators: Proper regulator selection ensures the safety, purity, flow-rate and pressure stability of gases delivered to an instrument. In general, most GC gas streams require a dual-stage regulator. I highly recommend dual-stage stainless steel high-purity regulators for carrier, make-up and other working gas supplies for sensitive detectors such as electron-capture detectors and mass spectrometers. These regulators are well worth the extra cost. High-purity regulators have been specially cleaned and contain non-contaminating materials that will not contribute extraneous substances to the gas stream. They also do a better job of preventing atmospheric gases from diffusing into the gas stream.

Cylinder fittings: In the US, gas cylinder pressure regulators follow a uniform convention, which is promulgated by the Compressed Gas Association (CGA, Chantilly, Virginia, website address http://www.cganet.com), for the types of high-pressure fittings that connect to gas cylinders. The fittings are designed so that incompatible gas cylinders cannot be connected to the same regulator. For the GC gases, hydrogen, air and carrier gases have different fittings. Other gases not normally encountered in GC such as oxygen or acetylene also have unique cylinder fittings that effectively prevent cross-use of regulators. The European Industrial Gases Association (EIGA) is the related organization in Europe. (For more information see http://www.eiga.org)

Never attempt to change the high-pressure cylinder fitting on a regulator or use an adapter to make a regulator work with gases other than those for which it was originally placed in service. Changing a regulator fitting can result in gas-line contamination in a gas chromatograph. With reactive gases, the result can be much worse. For example, oxygen regulators are specially cleaned to avoid internal combustion of oxidizable contaminants. A non-oxygen regulator pressed into oxygen service can quickly cause an explosion or fire.

Regulator installation: When installing a regulator on a high-pressure gas cylinder, first check the cylinder gas designation and ensure that the cylinder's fitting matches that of the regulator. Next, check the gas fitting seat in the cylinder for particulate or other contamination. If necessary, blow out the fitting with clean, dry compressed air. Never open the high-pressure cylinder valve in an attempt to clean the fitting seat — it's a very dangerous procedure that can result in injury. Remembering that some fittings screw on counterclockwise, thread the gas fittings together until finger tight, and then use a 12- or 15-in wrench to securely tighten the regulator to the cylinder. Don't use a larger wrench because the extra torque might deform the fittings. As with swaged fittings, apply no sealing or lubricating material to the cylinder or regulator fittings.

Unscrew the pressure-adjustment knob until the internal spring is no longer compressed and ensure that the regulator outlet valve, if one is installed, is closed. An outlet valve prevents air from leaking from an open regulator into the gas lines and it is a necessity for most GC gases. An additional in-line purge valve is useful for purging air from a reconnected gas line.

Place the gas cylinder with the pressure regulator positioned on the side opposite to you, slowly open the high-pressure cylinder valve, and allow the internal pressure to build up slowly in the regulator. Shut the valve immediately if an audible gas leak occurs. After the full pressure is established in the regulator, fully open the cylinder valve until it stops. This action will seat the top of the internal valve and prevent a leak around the valve itself. For gases other than air or nitrogen, use an electronic leak detector around the high-pressure fitting, the regulator gauge and the cylinder shutoff valve to ensure the absence of leaks.

Regulator testing: Gas regulators have a finite lifetime. Most regulators will last for years with normal GC use but each regulator should be tested after installation and periodically thereafter to identify potential premature failure.

When testing a regulator, first check the high-pressure gauge — it should read between 1800 and 2600 psig (12–18 mPa) for a new cylinder, depending upon the type of cylinder and gas. If the gauge reading is very low, then the cylinder is not full or the gauge is defective. If you obtain a low gauge reading, the best procedure is to temporarily install another suitable regulator to check the cylinder pressure. If necessary, replace the cylinder or the defective regulator. Never try to replace a gauge on a regulator or repair any other regulator component. Only a regulator manufacturer can do that.

Never try to replace a gauge on a regulator or repair any other regulator component.

Next, observe the outlet-pressure gauge for a few minutes with the outlet valve closed. You should see no observable pressure increase. If the outlet pressure increases when the pressure adjustment is fully withdrawn, then the regulator has a leak from the high-pressure side and must be returned to the manufacturer for repair or replacement. To complete checking the high-pressure side, close the cylinder valve and wait 2 min — the high-pressure gauge indication should not decrease. Any loss of high pressure with the cylinder valve closed also indicates a leak that will require repair or replacement.

With the outlet valve still closed, reopen the high-pressure cylinder valve completely. Adjust the regulator outlet pressure to its operating level, which is usually 40–90 psig (275–600 kPa) for GC gases. If the outlet-pressure gauge rises quickly to a high pressure or if the gauge fails to attain the desired level despite fully increasing the pressure adjustment, shut off the cylinder valve and replace the regulator.

At this point an in-line purge valve can be used to bleed out any air that might have entered the regulator. Open the outlet valve slowly and pressurize the connecting tubing. Bleed gas from the purge valve, if used, until any air is flushed out and then close the valve. The outlet-pressure gauge might drop momentarily, but it should settle back quickly to its set point. As a last step, check the dynamic operation of the regulator by momentarily shutting off the cylinder valve while the regulator is delivering flow. The high-pressure gauge will start to drop as the gas

Never mix ferrules, nuts and fittings of different manufacturers unless the supplier states specifically that its product is compatible with another supplier's fittings.

is consumed, but the outlet pressure should be steady as long as at least 2–3 times the outlet pressure remains on the high-pressure side. Restore the cylinder valve to its fully open position. Sometimes it's convenient to run this test while waiting for the gas lines to be purged of any remaining small amounts of air that entered during installation.

Tubing and fittings: As with all components in the GC supply gas stream, the connecting tubing and fittings must be free of contaminants and leaks, and they must be rated to withstand the highest possible pressure to which they could be subjected in the event of pressure regulator failure. A good estimate to use is at least twice the opening pressure of the safety relief valve in the downstream pressure side of the regulator; this pressure will be greater than the highest outlet pressure that the regulator is designed to deliver. Even so, higher-pressure transients are possible with failure of the high-pressure side of the regulator until the contents of the cylinder have been vented.

Plastic tubing: For the above reasons, GC installations should never use polymeric tubing or plastic fittings. Although these materials are suitable for many liquid chromatography (LC) applications, they are unsuitable for GC use for three reasons. First, polymeric materials can contaminate the gas stream. Atmospheric gases, namely water and oxygen, can diffuse into a gas stream, and the tubing can emit traces of plasticizers. Second, polymeric tubing and fittings could fail and burst at high pressures. Third, when they are routed behind a GC instrument, polymeric tubing and fittings can be exposed to the high-temperature air exhaust from a GC oven that is cooling down, and this exhaust could cause an immediate tubing failure or at least weaken a section of the tubing.

Aluminium tubing: I've seen a few installations that use aluminium tubing. Although possibly less expensive than copper or stainless steel, aluminium lacks the ductility of other metal tubing materials and it will rapidly develop metal fatigue cracks and failures unless mechanically constrained. The majority of GC gas supply installations will flex the connecting tubing during tank changes, and some gas chromatographs will flex external connecting tubing when their top covers are lifted. In addition, aluminium tubing does not fare well in swaged fittings that must be disconnected and reconnected.

Copper tubing: Copper is by far the most commonly used GC tubing material. Rolls of specially cleaned copper tubing are readily available from GC manufacturers and supply houses, making this the most convenient way to obtain suitable material. Copper tubing withstands the kind of flexing encountered in normal GC use. It can be uncoiled and coiled again easily for storage, and swaged fittings on copper tubing can be reconnected many times, if operators are careful to avoid overtightening the fitting and distorting the threads or ferrules. Copper tubing is best cut with a rotary blade tool designed for that purpose, and this tool can be purchased from any tool supply company. Never use a manual saw, pliers or diagonal cutters to cut tubing for GC purposes and never try to flex the tubing until it breaks from fatigue.

Stainless steel tubing: Many gas chromatographers prefer stainless steel tubing for critical applications. It is more rigid than copper and its spring properties allow it to better withstand limited flexing. Because of its hardness, however, stainless steel is more susceptible to leaks from minor imperfections on swaged fitting sealing surfaces. Stainless steel is more difficult to cut properly; it will quickly dull the rotary-type tools that can be used with copper. Instead, use a high-speed cutoff saw or other tool designed for use with stainless steel. Specialized deburring and dressing tools will prepare tubing ends that are square and free of scratches. After cutting, the tubing must be flushed with clean solvent to remove particulate residue and then thoroughly dried.

Fittings: Always use swage-type fittings for GC applications. Quick-disconnect, soldered, pipe compression or flared fittings are unsuitable. Permanently installed stainless steel tubing can be welded but it is more convenient to use discrete fittings instead for the smaller tubing sizes commonly encountered in GC.

Several manufacturers supply swaged fittings for GC applications. The use of one brand or another is a matter of personal preference, prior experience and perhaps most significantly, what type of fittings are on hand or already in use. Never mix ferrules, nuts and fittings of different manufacturers unless the supplier states specifically that its product is compatible with another supplier's fittings: the only result will be a leaking connection. In any one laboratory, it's a good idea to keep only one type of swaged fitting on hand to avoid a mix-up.

Try to match the fitting and tubing materials. The easiest way to remember is to match the colour of the ferrule, fitting and tubing. Use stainless steel for stainless tubing and brass for copper tubing. Brass fittings will also work with stainless steel because the softer brass ferrules will deform and make a seal but this arrangement could fail when reconnected several times. Stainless steel ferrules will not work as well with copper tubing because the ferrules will tend to crimp the tubing instead of forming tight seals to the inside of the receiving unions or bulkhead fittings.

Don't overtighten swaged fittings. In general, use two wrenches — one to turn the nut and one to restrain the union — and tighten a new fitting assembly approximately one-half turn beyond finger tight and then check for leaks and retighten if necessary. It's best to follow the individual manufacturer's recommendations for fitting tightening. For a reconnected fitting that's assembled onto an original union, only one-quarter turn may be necessary. Never try to force a fitting to seal by continuing to tighten it with a bigger wrench; you'll only ruin the nut and the union or bulkhead fitting. When in doubt, it's better to cut the tubing and make a new connection. Never apply any type of lubricant, sealant or polyfluorocarbon tape to the sealing surfaces of a swaged fitting — they're designed to seal with a close metal-to-metal contact. It is appropriate to use one layer of polyfluorocarbon tape on the threads of a bulkhead union in which the threads form the seal. Wrap the tape flat around the fitting once in the opposite direction of how the fitting will be installed.

Removing and Returning Used Cylinders

When the residual cylinder pressure drops to less than approximately threefold the regulator-outlet pressure, which is approximately 250 psig (1.7 MPa), it's time to replace the cylinder. Don't allow the internal cylinder pressure to go to zero; it will force the gas supplier to perform extra cleaning on the cylinder because they cannot assume it has not been contaminated. In addition, running down the cylinder pressure will cause the regulator's outlet pressure to first increase slightly and then drop off towards zero, which will cause retention-time and detector-stability problems. First, bring a new cylinder into the laboratory and then swap the old and new cylinders as described below.

The pressure regulator must be relieved of internal gas pressure before it's disconnected; otherwise a regulator could give a sudden burst of gas. First, turn the GC thermal zones off and allow them to cool. Turn off the detectors as well. Then, turn off the high-pressure cylinder valve and allow both the high-pressure and outlet-pressure gauges to approach zero. It might be necessary to bleed off gas at the gas chromatograph by increasing a flow or pressure setting temporarily. Next, turn the pressure adjustment on the regulator fully off and close the regulator outlet valve. Turn the GC flow and pressure settings to zero to prevent air from back-diffusing into the gas lines and filters. Finally, loosen and remove the regulator from the gas cylinder and install the cylinder cap.

Carefully secure the regulator while exchanging the cylinders. A small strap or chain works well to hold it onto a neighbouring device, or just place it on a flat surface. Don't leave it hanging by the connecting tubing — it will stress the tubing and could allow the regulator to fall some distance and be damaged.

If a regulator will be removed from use, even for a day, it should be detached from the connecting tubing and stored in a dust-free environment. If gas filters are in-line, be sure they are filled with gas and then seal the filters' inlets and outlets before exposing the gas supply lines to open air.

Return used cylinders promptly. Keeping empty cylinders on hand in a laboratory will accumulate cylinder demurrage charges and waste space that could be put to better use.

Conclusion

Laboratory workers can ensure high-pressure gas cylinder safety by following a few simple procedures and installation guidelines. Proper cylinder restraint, appropriate regulator installation and operation, and suitable connecting tubing and fittings will all yield improved safety and better GC results. Cylinder transportation seems to be the most hazardous part of gas handling in laboratory environments. Perhaps the hazard is caused by simple carelessness and a rush to get the instruments up and running again, but the potential cost and effect of failing to follow safe procedures far outweigh the loss of a few minutes of productive laboratory time.

Reference

1. J.V. Hinshaw, *LC•GC Eur.*, **15**(12), 787–791 (2002).

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