Reprinted from *American Laboratory* September 2005

Ten Steps to Maintaining Gas Stream Purity

by Frank Kandl

Many problems in gas chromatography follow a cylinder change-out, prompting chemists to buy ever-higher purity grades of gas. Yet, problems such as elevated baseline noise, ghost peaks, and excessive column bleed are often the result of contaminants introduced by the gas management system, not the cylinder. It can be thought of as drinking clean water through a dirty straw. This application note discusses 10 steps to maintaining gas stream purity, yielding more consistent performance and operating savings.

Gas management systems

Gas management systems contain many components—gas source, regulators, tubing, fittings, purifiers, traps, joints, and valves—all of which can be a source of leaks and contamination (see *Figure 1*). It is relatively easy to contaminate a system and time consuming to regain a baseline. Contamination can contribute to column damage and add gas and labor costs from rerunning samples and longer downtimes. The problem becomes especially costly if users attempt to solve it by buying a more expensive gas grade.

Instead, users can build a better gas management system that will 1) contain and remove contaminants before they do damage, 2) provide a visual indication as to when contaminants enter, and 3) identify the contaminant. The following 10 steps are important to remember.

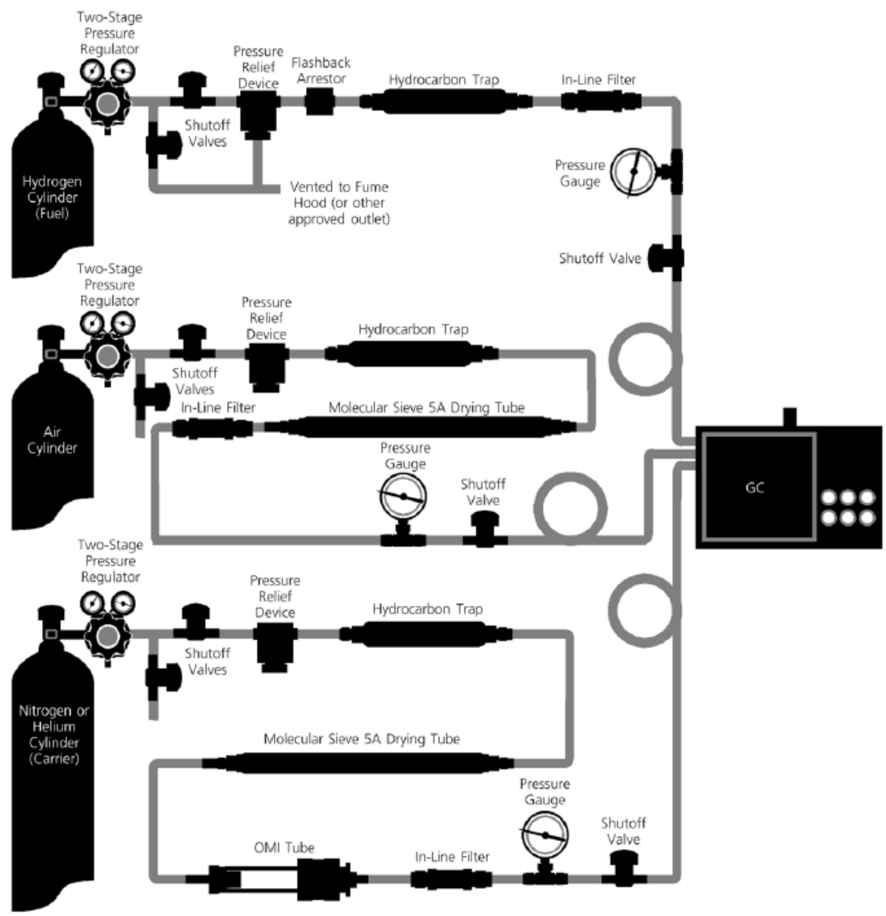

Figure 1 Gas management system schematic. Reproduced with permission from ***Sigma-Aldrich Co.,*** *Bulletin 898, T196898, 1996, p. 15.*

1. *Choose the right gas grade.* There is a myth that higher-purity gas will improve analytical results. If the gas, no matter how pure, travels through a dirty system, it becomes contaminated. If the system is clean, chromatographers may be able to improve the baseline to detect at lower levels or switch to a lower purity level to save money.

To choose a purity level, the chromatographer must first determine what impurities can affect the process. The gas supplier provides an analysis for these contaminants, which should be reviewed prior to purchase. The three primary impurities that affect most GC systems are oxygen, moisture, and hydrocarbons. Oxygen can accelerate column bleed, reduce column life, and change retention times. In some applications, it can also cause ghosts or unexpected peaks. Moisture can reduce column life, shift retention times, and increase baseline noise levels. Hydrocarbons can also increase baseline noise, degrade analyte quantification, and cause ghost peaks. All three of these contaminants are present in the atmosphere; thus, preventing them from mixing with the cylinder gas, whatever the purity level, is critical.

2. *Avoid contamination during cylinder changes.* Contamination most commonly occurs when the connection between the gas supply and the regulator is broken. When the cylinder is disconnected from the regulator, the system goes to atmospheric pressure, allowing atmosphere to enter. A properly designed system with the right components will minimize, contain, and remove the contaminants. The first line of defense is the connection designated by the Compressed Gas Association (CGA). This CGA nipple should incorporate a high-purity, nonlubricated check valve in its nose to keep the system pressurized during the cylinder change-out. Then, a

block-and-bleed valve can contain and remove the small amount of contaminants that enter, while the downstream system maintains pressure.

3. *Use the right regulator*. Another myth is that any regulator with a stainless steel diaphragm is suitable for analytical service. That is only half right. High-purity regulators use stainless steel diaphragms since they do not absorb contaminants. General-purpose regulators use neoprene diaphragms, which can absorb and then emit gas contaminants into the gas stream unpredictably.

Body construction—bar stock or forged body—is actually more important. A bar stock regulator has a small internal volume and a straight gas path. If contaminants do enter, it is easy to predict when they would emit. By contrast, a forged body has a large internal cavity with no direct path, allowing contaminants to become trapped and emit unpredictably. Only regulators made of bar stock, cleaned for chromatographic service, with a stainless steel diaphragm, should be used for GC systems. Regulators should never have lubricants or Teflon® (**DuPont,** Wilmington, DE) tape on the CGA nut, since these cause leaks and contamination as well. The user should consider whether a two-stage regulator is needed to maintain a constant pressure in the system. A single-stage regulator will require frequent adjustment as cylinder pressure decreases.

4. *Mount cylinder regulators correctly*. Most regulators weigh approx. 8 lb and are not easy to handle during a cylinder change-out. If the weight is unsupported, tubing can become

Figure 2 *Regulator mounting*.

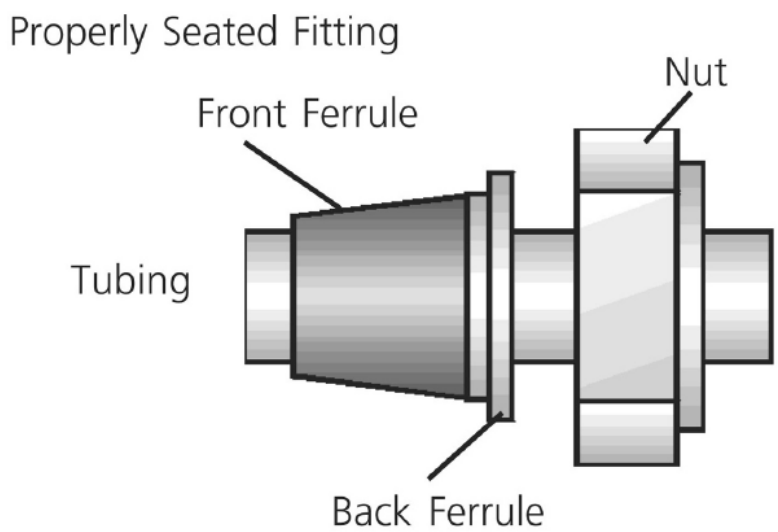

Figure 3 *Joint tightening. Reproduced with permission from* ***Sigma-Aldrich Co.,*** *Bulletin 898, T196898, 1996, p. 11.*

kinked, cracked, broken, or dislodged, causing a leak. A simple remedy is to use a wall-mounting bracket to support the regulator (see *Figure 2*).

5. *Use the proper tubing*. Chromatographically cleaned and "passivated" stainless steel or copper tubing should be used. Problems arise when mixing metals. Brass regulator fittings, for instance, will not achieve a tight seal on stainless steel. Many users purchase the correct tubing, but contaminate it during installation. For example, applying oil to the tubing cutter may make it easier to use, but may also introduce hydrocarbons into the system. Cutters must be free of lubricants, or the tubing must be recleaned after cutting.

6. *Make joints correctly*. For compression fittings, a two-piece ferrule made of the same material as the tubing should always be used. Brass ferrules on stainless steel tubing, for example, will not create a tight seal. Another misconception is that a loose fitting will only allow gas to leak out of the system. This is true when the system is in a static state. However, when gas is flowing, the leak generates a vacuum that can suck in atmosphere, which contains oxygen, moisture, and hydrocarbons. Conversely, many people tend to overtighten ferrules, which can crush them, creating a leak that permits these same contaminants to enter. The ferrule should be tightened only to the manufacturer's specification (see *Figure 3*). Also, if the system has brazed joints, they must be made using the fluxless brazing method. Orbital welding is a viable alternative since it creates a metal-to-metal fusion joint that has no filler metal. Pipe thread joints must only use Teflon tape without a lubricant or sealer.

7. *Use the right flexible pigtails*. Flexible pigtails are convenient, but those with a Teflon core are porous enough to allow small molecules of helium and hydrogen to diffuse through the wall. This can waste more than 10% of the gas. Teflon can also off-gas contaminants. Instead, leak-tight, flexible pigtails with internal stainless steel corrugated bellows should be used.

8. *Select the right valves*. Valves are another common contamination source. Three common valves in gas delivery systems are ball valves, needle valves, and diaphragm packless valves. Ball valves are popular because they provide a visual indication of whether they are opened or closed. However, in helium and hydrogen, these do not provide a positive shut-off, and they require lubrication, which may introduce

Figure 4 *Analytical changeover system.*

contaminants to the system. Needle valves provide flow control, but may introduce contaminants from lubricants and the potential off-gassing from the packing. The best valves are diaphragm packless valves, since they use multiple metal diaphragms, no lubrication, and are capable of passing helium leak tests.

9. *Install a cylinder changeover system.* Many chromatographers believe that a cylinder change requires 4 hours to recover a baseline. This recovery time is usually due to contaminants entering the system. Even with the safeguards described above, a cylinder change-out can take 30 min or more. Users too often replace the cylinder when convenient rather than when necessary. They will change a cylinder with more than 500 psig of pressure remaining on a Friday afternoon to avoid the expected downtime.

In contrast, an automatic changeover system allows for uninterrupted service, eliminating waste and downtime. When one cylinder is depleted, the system switches to the other side, allowing the empty to be returned with a minimum of residual (150 psig). When selecting a changeover system (see *Figure 4*), the chromatographer should look for traits similar to a regulator—bar stock construction, nonlubricated, check valves, and block-and-bleed valves.

10. *Use purifiers only as a backup*. There are three types of purifiers: surface absorbent, chemically absorbent, and chemically absorbent indicating. Purifiers safeguard against contamination. They are not a practical method for cleaning gas. In fact, using some types can actually introduce impurities at unpredictable moments.

Surface absorbent purifiers are the worst choice. When they become 40–60% saturated, the internal velocity increases, and impurities introduced during a cylinder change-out can propel previously captured contaminants into the gas stream. When completely saturated, the purifier may release most of its contaminants at once, requiring a long and arduous time to clean the column.

Because chemically absorbent purifiers lock up contaminants, they cannot become dislodged later. However, if the purifier is nonindicating, it is difficult to measure its saturation level. At some point, the medium will stop working, permitting contaminants to pass through unabsorbed. In-line purifiers also need valves on both sides to prevent contaminants from entering the system during a change-out.

An indicating purifier changes color when it chemically absorbs impurities. This color change gives the user a visual indication that impurities have entered and of what type, and when to change the purifier. In some indicating designs, the purifier changes color from the top down when impurities enter from the gas source, and from the bottom up when it detects contaminants from downstream. A quick-change base with integral check valves can also protect the gas stream when changing a spent purifier cartridge.

Conclusion

The right gas delivery system will improve consistency, limit downtime, and reduce costs. Chemists should specify components and construction methods to deliver clean gas and preserve the integrity of their systems. Ultimately, even the purest gas is wasted if it passes through a dirty straw.

Laboratories should seek suppliers that understand chromatography and will work with them to improve their systems. For facilities requiring larger amounts of gas, microbulk and bulk supply options can ensure a consistent supply of high-quality gas, with fewer opportunities to introduce contaminants. Use of these larger modes also eliminates the headaches associated with managing a large number of cylinders. Ultimately, maintaining gas stream purity enables chemists to focus on the work they do best.

Mr. Kandl is National Technical Manager, Specialty Gas Equipment, ***Airgas, Inc.,*** *tel.: 800-939-5711; e-mail: frank.kandl@airgas.com; website: www.airgas.com.*

Airgas®