



GC

Connections

This month, John Hinshaw explains some of the benefits and pitfalls of computer-controlled pneumatics systems. Their alarms can alert chromatographers to dangerous or damaging problems, but their capabilities can be compromised by a lack of operator training or understanding.

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An Alarming Situation

Modern gas chromatography (GC) instruments often include computer-controlled pneumatics options that provide a wealth of benefits. These subsystems regulate instruments' gas flows and pressures with greater precision and accuracy than manually adjusted pressure regulators, flow controllers, and needle valves. In addition, they provide users with the convenience of keyboard entry for pneumatic settings, and they allow external data systems to modify settings as required.

As with all sophisticated devices, computer-controlled pneumatics systems require some additional operator skills and knowledge to obtain the most successful results. Any pneumatic system, whether it be manual or electronic, should be calibrated and checked regularly; an electronic system has an increased possibility of unintended errors. This potential problem exists because a computer-controlled pneumatics system can indicate that all is well when the readings actually refer to erroneous settings or calibration errors unknowingly committed by chromatographers. For example, if a flow controller is calibrated for helium carrier gas but then subsequently is operated with nitrogen, the flows delivered by the controller will be inaccurate unless the pneumatic system is set for nitrogen operation. Even then, an operator should check the controller calibration with nitrogen gas for the most accurate operation. As a second example, pressure transducers' zero set points in a pneumatic control system will drift gradually over time or with thermal or mechanical stress. Without periodic transducer rezeroing, the actual column inlet pressure — as opposed to what the transducers indicate — will change and potentially compromise retention-time accuracy.

Computer-controlled pneumatics systems depend upon users to provide correct operating parameters such as column diameters and lengths or gas identities. If users fail to input the correct information, the computer-controlled pneumatics system will not function correctly — a good example of the Garbage In, Garbage Out principle.

Even if users have calibrated and checked all the computer-controlled pneumatics systems, some additional system features still can cause a degree of consternation if they are misunderstood. I am referring to the alarm indicators built into computer-controlled pneumatics systems; these indicators safeguard systems from damage caused by lack of column flow or, more dramatically, from a potential hydrogen explosion.

Guarding Against Explosion

The most dramatic aspect of the computer-controlled pneumatics alarm system is that it forces users to consider the possibility of a hydrogen explosion, which reminds me of apocryphal stories of exploding instruments and images of blown-out GC ovens. In fact, although the risk of an explosion is real, it is smaller than neophyte operators might believe. Many GC instruments do consume hydrogen as a combustion or carrier gas, but the explosion risk must be assessed for each situation. Of course, instruments that use no hydrogen have no such explosion risk. However, the second alarm function — to protect system components and the column — presents additional good reasons not to disable it.

Some situations might preclude hydrogen use because hydrogen isn't permitted in the vicinity of the instrument. Many process-control gas chromatographs, for example, are installed in hazardous chemical production areas where safety rules take precedence over the possible desirability, for chromatographic reasons, of hydrogen carrier gas or combustion-type detectors. In those situations, the presence or absence of a computer-controlled pneumatics alarm would make little difference. Sometimes an independent, explosive-gas sensor installed in a GC oven can safeguard the instrument and surrounding area by cutting off hydrogen flow at the supply tank.

Combustion gas only: Some laboratories restrict hydrogen use to combustion detectors only and do not permit it as a carrier

gas. When hydrogen is used only for combustion gas — in flame ionization detectors, for example — the explosion risk is low. The primary cause of very rare explosions in this situation is the combination of a column disconnected or broken at the detector end, a blocked detector vent, and combustion-gas pneumatic failure or incorrect setup, which can cause hydrogen gas to flow back into the GC oven. Even then, the hydrogen gas inflow must be great enough

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to create an explosive mixture in air — greater than 4% volumetric concentration — and an ignition source must be present.

For most hydrogen-consuming detectors, including the flame ionization, flame photometric, nitrogen–phosphorus, and electrolytic conductivity detectors, normal hydrogen flow rates are low enough that even if all the hydrogen from two detectors were to flow into the oven, the concentration isn't likely to reach the lower combustible limit. Hydrogen diffuses quite rapidly, and modern GC ovens leak some air in and out by design; both conditions reduce the risk of an explosive buildup. Many older ovens, however, were designed to be as airtight as possible for perceived purposes of thermal regulation, and they are the ones that pose the greatest risk of hydrogen buildup. Unfortunately, they don't benefit from a computer-controlled pneumatics alarm system.

Reversed connections: The chances of a pneumatic system failure that sends large quantities of hydrogen into a GC oven are small. However, the chances that a chromatographer will make the simple mistake of connecting the hydrogen tank supply to the air inlet on the back of a gas chromatograph are much greater. I observed this error in action once in a sales situation — and no, it wasn't me. The popping sound

that chromatographers normally hear after lighting the flame ionization detector allegedly was supplanted by a very loud report, yet this sound didn't alert anyone to the problem. No one had thought to check the flow rates after installation because the instrument had been operating just fine at another location two days earlier. After I arrived on the scene, the salesperson leaned over the top of the instrument to point out some feature and a hole melted in his necktie immediately. The flame, burning with several hundred milliliters-per-minute of hydrogen, was invisible, and it extended several inches above the detector vent. Always verify combustion-gas flow rates with an external flowmeter each time an instrument is moved or gasses are hooked up. In the reversed-connection case, the measured air flow, which really is hydrogen, will be much higher than the computer-controlled pneumatics set point. Any discrepancy such as this one should alert operators that something is amiss, and they should immediately shut off the gas flow and examine the entire system setup carefully.

Ignition sources: Combustible gas detectors provide a couple of ready ignition sources. The first, of course, is the flame that burns in the detector. In single-detector systems, this flame isn't a problem; if the flame is burning, then the hydrogen is being consumed. In a dual-detector system, however, one detector's flame might ignite hydrogen leaking from the other detector. More often, the main ignition source is the act of lighting the flame itself. Thus, operators must be very sure that normal gas flows are exiting from a flame detector before igniting it. It is a dangerous practice to turn on the gases and attempt to ignite a detector without first checking for the correct flows. If the gases aren't calibrated and set correctly or if the detector has some other problem, then it's likely to fail to light or maintain a steady flame. In those situations, the oven could have a significant hydrogen leak and ignition could propagate a potentially injury-causing explosion.

Several models of gas chromatographs feature automatic flameout detection and reignition. This convenient feature is designed for instruments that largely run unattended, but it raises one critical question: Why did the flame go out in the first place? If the flame has extinguished, then potential hydrogen flow problems should be investigated before reigniting it.

Hydrogen as carrier gas: Hydrogen has two significant advantages as a carrier gas for open-tubular or capillary columns. It exhibits nearly optimum column efficiency over a wider range of gas velocities than does helium or nitrogen, and it requires lower pressure drops to produce optimum velocities, which makes it especially well suited for longer, narrow-bore columns. A third advantage in some areas is its ready availability and lower cost compared with helium.

With hydrogen as the carrier gas in older manual fore-pressure-controlled inlet systems, an explosive mixture can collect in the GC ovens if the columns are broken at the inlet connection. These carrier-gas controls try to deliver a constant pressure to the column without regard to the flow rate. If no column is present, then flows greater than 1 L/min could enter the oven and create an explosive mixture. One approach to limiting hydrogen flow in this situation is to use a restrictor in-line between the hydrogen tank and the gas chromatograph. As hydrogen flows increase, the pressure drop across the restrictor also increases, which causes the supply pressure to the instrument to drop and eventually limits the flow rate.

In a back-pressure-controlled system, carrier gas is delivered to the inlet through a flow controller that regulates the total split flow. If a column breaks at the inlet, the flow controller limits the total flow rate and helps reduce the likelihood of an explosive buildup. Most computer-controlled pneumatics systems use this type of pneumatic arrangement for split-splitless inlets.

Computer-controlled pneumatics systems can detect these fault situations and can take action to limit the potential hazard. If the electronic pneumatic system is unable to establish the set column-inlet pressure after several minutes, it activates an alarm. Similarly, the alarm is turned on if the set split flow rate is unattainable or if a system detects related defects in any of the other pneumatic zones, including the detector combustion gas supplies. If the situation is not remedied within a few minutes, the alarm condition will cause the electronic pneumatic system to cut gas flow entirely and cool the oven. Some systems go so far as to completely remove power from all of the active components in the GC system in this situation. In any case, any gas that has accumulated will dissipate quickly with the hydrogen source turned off.

Protecting Instrument Components

The alarm functions of a computer-controlled pneumatics system that limit a potential explosive buildup should not be disabled simply because hydrogen is absent as a carrier or combustion gas. Even without hydrogen, the second function of the alarm system — to protect instrument components — is well worth keeping the system functional.

If a column breaks or the carrier-gas tank empties or if carrier cannot be delivered to the column for any other reason, shutting down the instrument might save the column from exposure to air and heat that will cause eventual destruction. By cooling the oven and, if possible the inlet and detector, after a pneumatic alarm event, a column is not heated for prolonged periods while exposed to the air.

The pneumatic alarm also protects detectors that are air-sensitive such as thermal conductivity detectors. The filaments inside thermal conductivity detectors operate at temperatures that are high enough to cause significant filament oxidation with exposure to air. Prolonged exposure leads to a loss of sensitivity and eventual filament failure. When an alarm occurs in an instrument with a thermal conductivity detector, the alarm shutdown will remove power from the filaments and limit the damage.

Avoiding False Alarms

As is the case with any alarm system, false alarms happen with computer-controlled pneumatics systems. The result typically is more irritating than anything else. In those cases, the pneumatic alarm starts beeping for no apparent reason and eventually shuts the GC system down. After a few false alarms it's tempting to shut the alarm system off, but operators can avoid most false alarms with a little care. Operation outside of normal parametric limits is the primary cause of false computer-controlled pneumatics alarms. In particular, two specific situations can cause false alarms, and both of them involve split-splitless inlet pneumatics.

Too much split flow: Inlet splitters are designed to conduct large split flow rates, but physics requires them to engender a finite pressure drop, which can be as much as 2–3 psig with split flows of 300 mL/min or greater. This natural back pressure can appear at the inlet pressure transducer in some designs. It will cause a false alarm if the inlet pressure set point is lower than

the natural pressure, because the pneumatic system cannot bring the carrier pressure level to less than the set point. This situation occurs most often with columns that have inner diameters of 530 μm or greater, especially when they are operated with the split injection system in a flow-controlled mode. These columns require very low pressure drops, often less than 1–2 psig, to deliver their optimum flow rates of 2–5 mL/min.

At the same time, high split flow rates are needed to attain split ratios as low as 100:1. As a result, the split system has a natural pressure drop of several pounds — due to the high split flow rate — while trying to achieve a column pressure drop that is significantly lower. The result is a computer-controlled pneumatics alarm. In this case, the alarm should be heeded because it indicates the column pressure drop is higher than the set point. This particular situation also gives credence to those of us who prefer not to install wide-bore columns in a split-inlet system.

Not enough split flow: The obverse of the above situation occurs when operators attempt to use a split-inlet system at very low split flow rates with a wide-bore column. This situation often occurs when attempting to analyze trace-level samples with a splitter. Lower split ratios place more material on the column and produce larger peaks. However, if the split flow rate is less than the sum of the desired column flow rate and the septum purge flow, then the inlet system will be unable to attain the set point pressure. For example, if a wide-bore column has a set flow rate of 5 mL/min and the septum purge flow is fixed at 5 mL/min, then the incoming split flow must be significantly greater than 10 mL/min. If not, the inlet system is starved of carrier gas — there's not enough to go around. The remedies for this situation are, first, to avoid attempting split injection with a sample that would be better served with splitless or on-column injection, and, second, to avoid using wide-bore columns in a split-inlet system.

Conclusion

Computer-controlled pneumatics offer chromatographers improved performance, convenient operation, and some protection from both explosive and damaging events. Operating these systems, however, requires a degree of specialized training and a good understanding of their capabilities and limitations. A computer-controlled pneumatics system's ability to flag potentially dan-

gerous or harmful situations and to enact a safe recovery before damage occurs is a great asset. But if that capability is disarmed because of a lack of operator training or understanding, then its usefulness is lost. As with any complex system, the computer-controlled pneumatics alarm capability has limits outside of which it will not operate properly; these situations nearly always cause false alarms that err on the side of safety rather than convenience.

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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>.

