Nitrogen: The Most Cost-Effective Makeup Gas for Gas Chromatography

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Abstract

Helium has been used as a makeup or carrier gas in the gas chromatography process since the 1950s-1960s. However, there are several major disadvantages to helium: it is expensive and the supply can fluctuate, depending on natural gas production and national supply-and-demand factors. Helium is also only available in tanks. Nitrogen, on the other hand, performs just as well as helium as a carrier gas and is far less expensive. The supply of nitrogen gas is unlimited and can be separated from air, directly in the laboratory, using an in-house gas generator, which vastly improves convenience and lab efficiency. Nitrogen also better defines the shape of the flame in the flame ionization detector (FID), enhancing sensitivity. Because of these key advantages over helium, more laboratories are switching to nitrogen gas for their gas chromatography needs.

Introduction

The makeup gas (carrier gas) in the gas chromatography process transports the ionized sample into the chromatograph. The most important property of the carrier gas is that it cannot react with the compounds being measured. Helium, an inert gas, has been traditionally used for this purpose for over 50 years.

Helium is generated by the radioactive decay of thorium and uranium. Helium is found in trace levels in the atmosphere and higher levels in natural gas (up to 7 percent by volume), from which it can be isolated by fractional distillation. Because helium is a relatively rare yet strategic commodity, the U.S. Bureau of Land Management (BLM) maintains a strategic stockpile to provide a reliable supply. Only a finite amount of helium is available for industrial/commercial purposes and is dependent on the extraction of natural gas. Over recent years, however, the drawdown on this reserve has been significantly greater than its replenishment, resulting in limited availability and higher cost.

Even though natural gas reserves have increased dramatically across the U.S. because of hydraulic fracturing of oil-shale deposits, sale of helium is still controlled by the U.S. government. The amount of helium used for gas chromatography is a very small percentage compared to the overall demand for helium; however, if demand from major consumers (for example, the medical industry requires helium for MRI-superconducting magnets) increases, there could still be supply restrictions.

Therefore the price of helium has steadily increased over the last few years. The BLM set the price of helium at $95/1Mcf [1,000 cubic feet] in 2014, a 13 percent increase over the $84 price in the fall of 2013.

A Shift toward Nitrogen

Because of the disadvantages of using helium as a carrier gas, more chromatographers are turning to nitrogen. Nitrogen is a nonreactive gas and performs just as well as helium as a carrier gas. Nitrogen is abundant in the atmosphere (78 percent of air is nitrogen), which makes it easy to provide an unlimited supply of high-purity nitrogen through fractional distillation of air, at a much lower cost (about $30/1Mcf). Nitrogen also improves the shape of the flame in the flame ionization detector (FID), which enhancing the sensitivity.

Nitrogen is commonly delivered in high pressure tanks—labs may not give this much consideration (especially if they have recently switched from helium tanks and are using the same supplier for nitrogen). What they don't realize is that nitrogen can be easily generated in-house from compressed air, with no need for heavy nitrogen tanks—virtually eliminating any supply problems. The initial investment in a nitrogen gas generator is recovered in about a year's time. After that, the cost of nitrogen is essentially free (the only related costs are electricity consumed and routine maintenance).

Generating nitrogen from an in-house generator (for example, the Parker Balston Model MGG-2500, Figure 1) requires seven stages:

1. Pre-filtration. A high-efficiency coalescing filter is used to remove water, oil, and particulate matter (to 0.01 μ) from compressed laboratory air to avoid damaging downstream components. The water and oil are removed from the filter by an automatic drain.

2. Adsorbtion of volatile organic compounds. Activated carbon is placed upstream of the catalyst to remove trace amounts of halogenated hydrocarbons and other volatile organic compounds that could

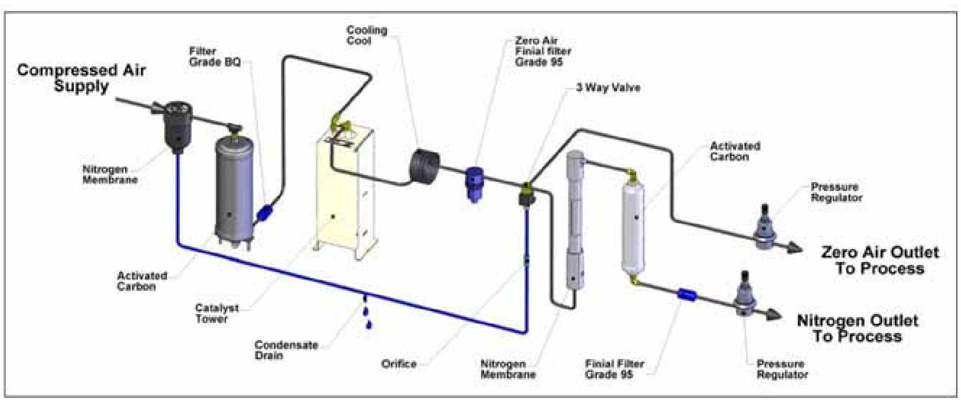

Figure 1: Design of an in-house nitrogen generator for makeup gas and zero air for a flame ionization detector for gas chromatography.

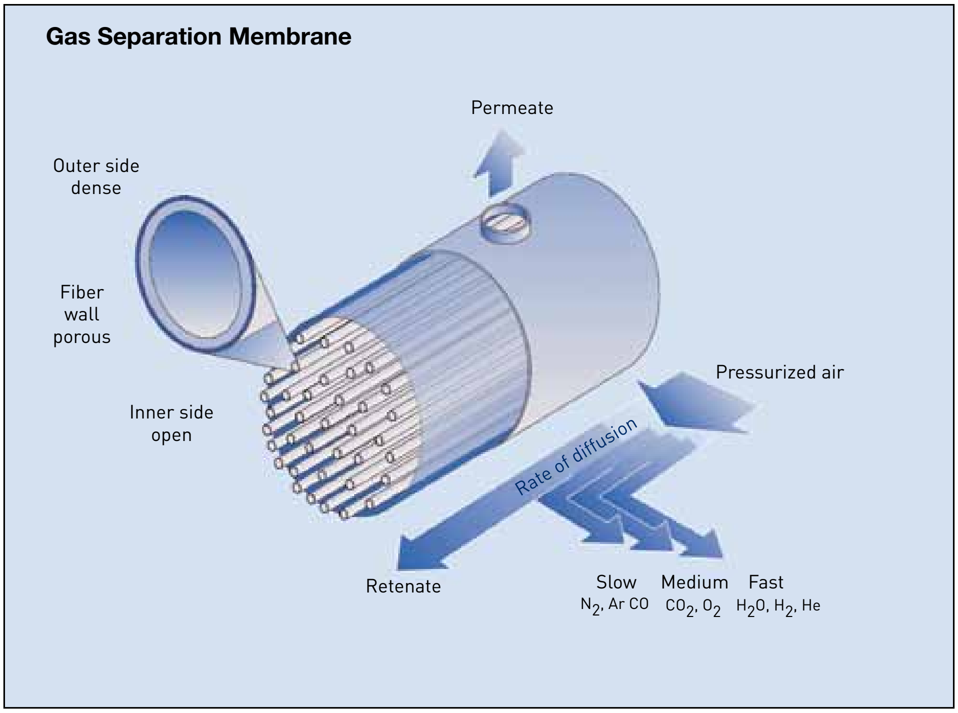

Figure 2: Separation of nitrogen via a semi-permeable membrane.

poison the catalyst. If halogenated hydrocarbons are present in the compressed air at a level exceeding 5 ppm, an auxiliary activated carbon scrubber can be used to avoid poisoning the catalyst.

3. Particulate filtration. A BQ-grade filter (99.99-percent efficient at 0.01 µ) is placed after the activated carbon filter to ensure that activated carbon particles are removed from the stream.

4. Hydrocarbon removal. A cartridge heater in a stainless-steel vessel with a proprietary catalyst blend oxidizes the hydrocarbons in the air into CO2 and H2O, resulting in a concentration of less than 0.05 ppm as methane.

5. Cooling. A copper coil allows the makeup gas to cool after passing through the heated catalyst module.

6. Filtration. The cooled gas is passed through an ultra-high-efficiency membrane to remove particulate contamination in the gas from the catalyst module.

7. Nitrogen purification via a semi-permeable membrane. A hollow-fiber membrane separates the nitrogen¬ from the other gases in the compressed air. Air flows through the tube as shown in Figure 2. Gasses that permeate at a rapid rate (CO2, O2, H2, H2O, He) are removed from the compressed air at a higher rate than nitrogen. A single-membrane fiber with a very small internal diameter and a nitrogen module with a large number of fibers bundled together provide a large surface area and generate a high nitrogen output.

Advantages of In-House Gas Generation

Nitrogen produced from an in-house generator is 99.9999-percent pure with respect to hydrocarbons (measured as methane) and 99-percent with respect to oxygen. In addition to providing nitrogen, an in-house generator also produces zero air (containing <0.05 ppm of hydrocarbon, measured as methane) to support the FID flame, eliminating the need for any cylinders to operate the FID.

The purified makeup gas is directly ported to the detector to provide gas that is available at all times, with no user activation required. The generator provides warnings if the air supply falls, excessive air flow is observed, or the temperature of the catalyst heater is incorrect.

Other advantages of nitrogen in-house generation include:

1. Safety. The in-house generator is hard-plumbed directly into the gas chromatograph and delivers nitrogen at a flow and pressure that meets the needs of the detector. The maximum pressure at the outlet is dependent on the inlet air pressure; the maximum flow rate is 400 mL/minute. In contrast, when a tank is used, the maximum pressure approaches 2000 psi. There is also risk involved in transporting a tank from the storage area to the laboratory and connecting it to the chromatography system. Serious personal injury and/or damage to the facility could result if control of the tank is lost during the transportation or installation. Also, if the tank valve is damaged, a large amount of nitrogen gas could escape into the laboratory, potentially causing an asphyxiation hazard.

2. Convenience. Since the in-house generator is hard-plumbed into the gas chromatograph, the makeup gas is available on a 24/7 basis. In contrast, when tanks are used, the operator must monitor the level of gas to ensure that there is a sufficient amount to perform the desired analyses (especially if an overnight series of analyses is planned).

3. Cost. An in-house generator can provide makeup gas at a considerably lower cost than tank gas. The Parker Balston Model MGG-2500 [insert link] generator, for example, requires 100 V/4 amps. If it is used on a 24/7 basis at a power cost of 10c/kwh, the approximate cost per day is $1.15. In contrast, when tank gas is used, the facility must bear the direct cost of the tank and demurrage charges. In addition, using tanks involve incidental costs such as the time required to obtain and install a tank, ordering new tanks, maintaining inventory, and related activities. Although the actual cost savings from using an in-house generator depends on a broad range of factors, many users report the payback period is a year or less, and the overall operating costs can be reduced by 50 percent or more.

4. Environmental impact. The use of an in-house generator has a significant benefit to the environment because it uses a minimum amount of energy. In contrast, when gas tanks are used to supply makeup gas, air must be cooled for fractional distillation; once the purified nitrogen is obtained, it must be compressed and tanks must be transported from the supplier's site to the end user. Similarly, once a tank is emptied, it must be returned for refilling; transportation of tanks requires a significant amount of energy.

5. Elimination of contamination. An in-house nitrogen generator eliminates the possibility of introducing foreign materials when a new tank is installed. When a tank is used to provide nitrogen, the user must periodically break the connection between the supply and the detector to install a new tank, which could lead to the introduction of materials from the atmosphere that would have a negative impact on the FID measurement.

Becoming Lean

There is no question that in-house nitrogen gas generation saves money after the initial investment is recovered, typically within about one year.

What is perhaps most valuable about in-house gas generation is that it greatly simplifies the gas chromatography process.

An in-house gas generator does this by eliminating unnecessary steps—the core idea behind the "lean" movement, which has been so successful in maximizing productivity and reducing costs in the automotive industry. By eliminating wasteful steps, increasing efficiency, and lowering costs, a company can increase its competitiveness in the marketplace.

Many companies across a wide range of industries are intensely committed to creating a "lean" organization by eliminating unnecessary (and therefore wasteful) steps.

When a process is simplified, there is less chance that something will go wrong or delay the operation. This reduction in risk saves time and money. A lean operation is also easier to operate, reducing operator stress and improving worker satisfaction/productivity. Being lean also means you can meet changing client demands more quickly—a huge added value. Part of the cost savings that is realized by switching from helium to nitrogen gas generation can also be passed onto the client, if desired.

In-house nitrogen gas generation is a remarkably simple, reliable, and effective way to eliminate the use of costly helium in the gas chromatography process.

Consider some of the extra steps (costs) involved when using helium:

- Setting up and monitoring helium tank deliveries
- Negotiating and maintaining third-party contracts
- The time and labor required for storing and moving tanks
- Safety risks associated with handling heavy, highly pressurized tanks

Also, during operations, a helium tank must be constantly monitored to be sure there is enough helium; if not, time is lost if the tank needs to be switched.

Switching from helium to in-house nitrogen gas generation eliminates these time-consuming steps that add cost and risk to the gas chromatograph process.

Conclusion

Many companies simply accept using helium because this is the way they have always done gas chromatography.

The limited availability and increased cost of helium is prompting many chromatographers who use FID detectors to switch to nitrogen for makeup gas. Even though high-purity nitrogen is available in tanks, nitrogen generated directly in the lab provides a number of significant advantages, especially lower cost. An in-house generator also increases productivity and eliminates the hassle of dealing with deliveries and the safety risks related to handling gas tanks.

In summary, in-house nitrogen gas generation is a highly efficient and cost-effective way to increase the quality of your analytical operations, at significantly lower cost. It also gives you greater control over your operational efficiency, with faster results—an added value that clients appreciate and that makes your lab more competitive in the marketplace.

To learn more about nitrogen gas generators, visit www.parker.com/balston or call 978-858-0505 to speak with a Parker Balston engineer.

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