Optimum Generation of Makeup Gas for GC-FID

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The Flame Ionization Detector (FID) is commonly used for gas chromatography (GC) as it can detect almost all organic compounds. While Helium has been traditionally used as a make-up gas, its availability has decreased and its cost has increased. Many chromatographers now use nitrogen as a make-up gas as it is readily available, inexpensive, improves the flame shape in the FID, and enhances sensitivity. High-purity nitrogen can be generated by the fractional distillation of air and supplied in a high pressure tank or can be generated in-house from compressed air.

How Does an In-house N2 Generator Isolate N2 from Compressed Air?

The design of a typical in-house N2 generator (Parker Balston Model MGG-2500, Parker Hannifin Corporation, Haverhill, MA) is presented in Figure 1.

a) Pre-filtration: Water, oil, and particulate matter (to 0.01 µ) are removed from compressed laboratory air.

b) Adsorbtion of Volatile Organic Compounds: Activated carbon is used to remove halogenated hydrocarbons and other volatile organic compounds that could poison the catalyst. A filter (99.99% efficient at 0.01 µ) placed after the activated carbon removes carbon particles from the stream.

c) Hydrocarbon Removal: A heater and a proprietary catalyst oxidize the hydrocarbons in the air into CO2 and H2O resulting in a hydrocarbon concentration < 0.05 ppm (as methane). The air is cooled after passing through the heated catalyst module and passed through an ultra-high-efficiency membrane to remove particulate contamination.

d) Nitrogen Purification via a Semi-Permeable Membrane: A hollow fiber membrane separates the N2¬ from other gases in the compressed air. Air flows through the tube as shown in Figure. Gases that permeate at a rapid rate (CO2, O2, H2, H2O, He) are removed from the compressed air at a higher rate than N2. A membrane fiber has a very small internal diameter, so a large number of fibers are bundled together to generate high nitrogen output.

The N2 obtained from an in-house generator is 99.9999+% pure with respect to hydrocarbons (measured as Methane) and 99+% to O2. In addition, the system provides zero air (<0.05 ppm of hydrocarbon, measured as methane) to support the FID flame, eliminating the need for any cylinders to operate the FID.

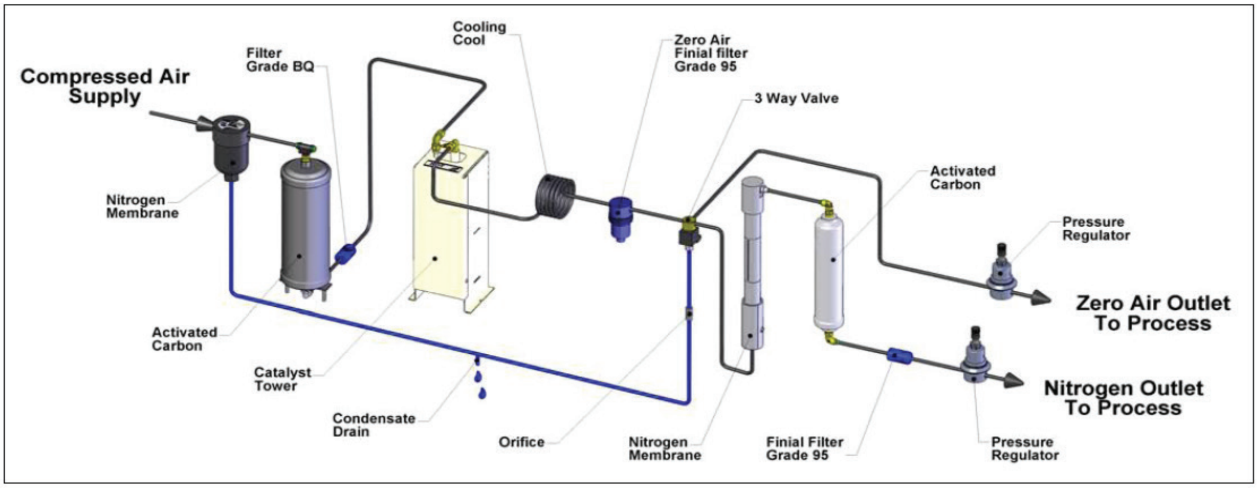

Figure 1: Design of a typical in-house Nitrogen Generator for Make-up Gas and Zero Air for a Flame Ionization Detector for Gas Chromatography

Benefits of an In-house Generator

a) Safety: An in-house generator is hard-plumbed directly into the GC and delivers N2 at a flow and pressure that meets the needs of the detector. The outlet pressure is dependent on the inlet air pressure and the maximum flow rate is 400 mL/min. When a tank is used (which may have a maximum pressure of 2000 psi), transporting it from the storage area and connecting it to the chromatograph could lead to serious personal injury and/or damage if control of the tank is lost. If the tank valve is damaged, a large amount of N2 could escape into the laboratory potentially causing an asphyxiation hazard.

b) Convenience: The in-house generator is hard plumbed into the GC so N2, is available on a 24-hour/7 day basis. In contrast, when a tank is used, the gas level must be monitored to ensure that it is sufficient to perform the desired analyses.

c) Cost: An in-house generator can provide N2 at a considerably lower cost than tank gas. The generator described above requires 400W; if it is used on a 24-hour/7 day basis at a power cost of 10c/kWh, the approximate cost is $1.15/day. In contrast, the cost of tank gas includes demurrage charges, the cost of the time required to obtain and install a tank, ordering new tanks, maintaining inventory, and related activities. Many users report that the payback period is a year or less and the overall operating cost is reduced by 50% or more.

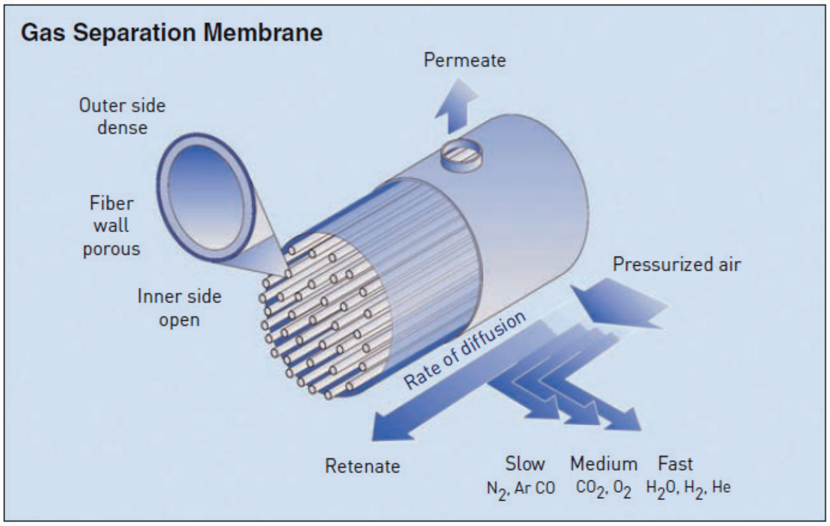

Figure 2: Separation of Nitrogen via a Membrane

d) Environmental: An in-house generator uses a minimum amount of energy. In contrast, when a gas tank is employed, air must be cooled for fractional distillation. Once the purified nitrogen is obtained and compressed, it is transported from the supplier's site to the end user (and returned for refilling), which requires a significant amount of energy.

e) Elimination of contamination: An in-house generator is permanently connected to the GC, minimizing the possibility of introducing foreign materials. When a tank is used, the user must periodically break the connection between the supply and the detector to install a new tank potentially introducing materials that could have a deleterious effect on the measurement.

Conclusion

The limited availability and increased cost of helium prompted many chromatographers who use FID detectors to switch to N2 for make-up gas. While high-purity gas tanks are available, the use of an in-house generator provides many significant advantages. An in-house generator eliminates the safety issues related to handling gas tanks and is much less expensive. Additionally, many users report that an in-house system pays for itself in less than a year. Once an in-house generator is installed and plumbed into the GC, it can provide the make-up gas with essentially no user interaction. Since the generator is directly plumbed into GC, the possibility of contamination is dramatically reduced, while foreign materials could enter the detector when tanks are replaced.

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