

SUPPLYING HIGH-PURITY GASES

MAKING A CASE FOR IN-HOUSE GAS GENERATORS **By Kim Myers and Peter Froehlich**

Selecting the most effective source for high-purity gases is a critical issue for laboratory managers. Zero air and hydrogen are used for gas chromatography with flame ionization detection (GC-FID), and nitrogen is used for high-performance liquid chromatography or gas chromatography with mass spectrometric detection (LC-MS or GC-MS). High-purity gases are used with other instruments: CO₂ free purge gas for Fourier transform infrared spectroscopy (FT-IR), highly purified nitrogen for inductively coupled plasma (ICP) systems, dry air for nuclear resonance spectroscopy (NMR) and hydrocarbon-free combustion gas for TOC analyzers.

Many laboratories employ in-house high-purity gas generators to supply the necessary gases, as they provide significant safety, convenience and cost advantages when compared to the use of tank gas.

In-house gas generators

Nitrogen, purge gas, ultra dry gas, source exhaust air and zero air can be obtained from compressed air, and hydrogen can be obtained by the electrolysis of water, using in-house generators. An in-house gas generator can be dedicated to a single instrument or used to supply multiple instruments (e.g., a hydrogen generator can provide fuel gas for 14 FIDs).

a) Generation of nitrogen

Nitrogen is generated from compressed air by the removal of oxygen, water vapor and particulate matter. The heart of an in-house nitrogen generator is a hollow fiber membrane

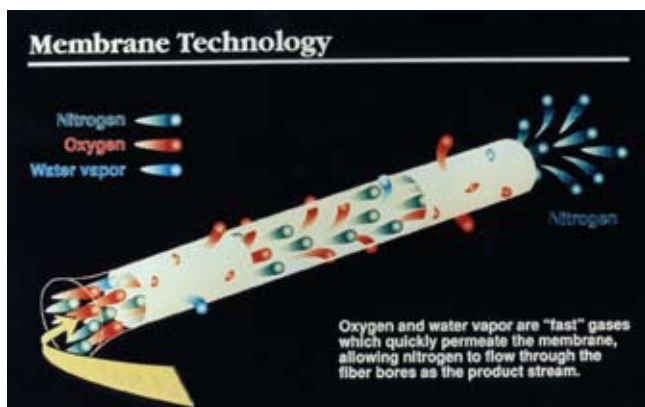


Figure 1. Operation of a Hollow Fiber Membrane



Figure 2. A Hollow Fiber Membrane Bundle

through which oxygen and water vapor permeate and escape through the sweep port while the nitrogen flows through the tube (Figure 1). A large number of fibers are bundled together (Figure 2) to provide an extremely large surface area.

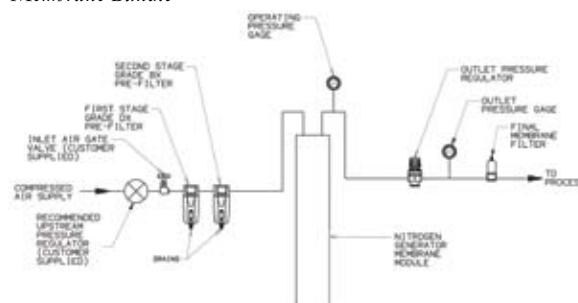


Figure 3. Schematic Diagram of N2-04 Nitrogen Generator (courtesy of Parker Hannifin Corp.)

The schematic of a typical nitrogen generator (Model N2-04, Parker Hannifin Corp., Haverhill, MA) is shown in Figure 3. Compressed air is filtered using a high-efficiency activated carbon filter to remove organic impurities, and a pre-filtration system is used to remove particulate matter >0.01µm. Air passes through a fiber membrane bundle to remove the oxygen and water, while the purified nitrogen is passed through another absolute membrane filter and delivered to the instrument.

The N2-04 nitrogen generator produces 99.5 percent pure nitrogen on a continuous basis (the purity of the nitrogen depends on the operating pressure and the desired flow rate, e.g., 3 L/min of nitrogen at 80 psi). The gas has an atmospheric dew point of -58°F (-50°C), contains no particulate matter >0.01 µm and no suspended liquids, and is hydrocarbon and phthalate free.

b) Generation of hydrogen by electrolysis of water

i) Using metallic electrodes

An inert anode and a cathode such as a bundle of palladium tubes are used to dissociate water (20 percent NaOH is added to enhance electrolysis). Hydrogen passes through the cathode while oxygen and other impurities collect at the anode, and hydrogen gas with purity in excess of 99.99999+ percent can be obtained.

ii) Using a proton exchange membrane

A proton exchange membrane (PEM) is an ionomeric (ionic polymer) membrane that conducts protons and is impermeable to hydrogen and oxygen. PEMs are used in fuel cells to create an electric current (and form water) from hydrogen and oxygen. When an appropriate potential is applied to a PEM in the presence of pure water (caustic NaOH is not required), water is dissociated to provide hydrogen and oxygen.

c) Generation of ultra dry air, purge gas, zero air and source exhaust air

Several techniques are employed to remove moisture, particulate matter and hydrocarbons from air to provide air with the desired purity. A coalescing compressed air filter, which consists of a matrix of borosilicate glass hollow fibers in a fluorocarbon resin binder, removes water, hydrocarbon lubricants and synthetic lubricants from compressed air. For instrument-grade air, two-stage filtration removes 99.99 percent of 0.01 µm particles and droplets and a three-stage filter system is used to remove compressor oil vapor. A heated catalysis module and high-capacity carbon absorption modules and coalescing filters remove very light methane-based hydrocarbons.

d) In-house multi-gas generating systems

In-house generation of nitrogen and purified air from compressed air is performed with the Source LC/MS Tri Gas Generator (Figure 4), which includes coalescing pre-filtration with timed solenoid drains, self-regenerating compressed air dehydration membranes, a proprietary heated catalysis module, self-regenerating nitrogen retentate membranes, high-capacity high-sensitivity carbon absorption modules and matched final

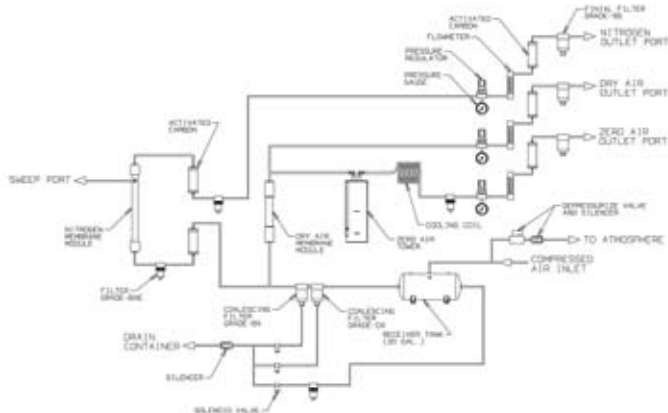


Figure 4. Schematic Diagram of Tri-Gas Generator (courtesy of Parker Hannifin Corp.)

Benefits of in-house gas generators

In-house gas generators provide significant benefits to the laboratory, including a dramatic improvement in safety, an increase in convenience and a lower cost.

Minimizing safety hazards

An in-house generator is considerably safer than tank gas; only a small amount of gas is stored at low pressure at a given time and it is ported directly to the instrument. As an example, the TriGas Generator generates 10L/min of nitrogen at a maximum pressure of 80 psig, 23 L/min of source air at a maximum pressure of 110 psi and 8L/min of exhaust gas at a maximum pressure of 60 psi. If a leak occurred, only a small quantity of nitrogen gas would be dissipated into the laboratory.

In contrast, serious hazards exist if nitrogen is supplied using a high-pressure gas tank or a liquid tank. If a full tank of nitrogen were suddenly vented into the laboratory, up to 9000 L of gas would be released, displacing laboratory air and reducing the breathable oxygen content.

“IN-HOUSE GAS GENERATION ALLOWS FOR CONTINUOUS OPERATION.”

An in-house gas generator also eliminates the possibility of injury or damage from the transportation and installation of a gas tank. A heavy gas tank can be a hazard to staff and facilities if the valve is compromised during transport (in many facilities, specially trained technicians replace gas tanks). A leaking hydrogen tank could lead to an explosion. When a Dewar flask or a high-pressure liquid tank is used to supply nitrogen, a leak or spill could lead to frost burns.

New construction or laboratory expansion

An important factor when considering new construction or expanding an existing laboratory is the gas requirements for the instruments being used. It is important to select a supply, manifold and purification system to ensure that the gases supplied will meet your requirements for pressure, volume and purity. Architects and designers will typically allocate space for cylinders or other gas storage options (i.e., Dewars or bulk systems). For many installations, a gas generator will provide a superior gas supply with higher purity for a lower operating cost and will require less floor space. In addition to the need for less lab space, less space for storage of back-up and empty cylinders is required.

Maximizing convenience

An in-house gas generator can supply gas on a 24-hour, seven-days-a-week basis with no user interaction (other than routine annual maintenance). Dr. Chris Ransom, a chemist at MedTox Scientific, a toxicology company in St. Paul, MN, reports that they have used in-house nitrogen and zero-air generators with GC systems and HPLC with MS for over ten years, with little or no maintenance, except for periodic changing of filters.

In contrast, when tank gas is employed, the user must monitor the level of gas in the tank and ensure that there is sufficient gas available for the desired analyses. The in-house system eliminates the need to retain extra tanks; when it is necessary to get a replacement gas tank, the chromatographer may need to find an individual who is qualified to handle the tanks. Tanks are typically stored outside in a remote area for safety reasons, and replacing tanks can be a significant inconvenience, especially in inclement weather. In addition, a pressurized tank could be a significant hazard if the laboratory is located in a seismic zone.

If a gas tank must be replaced during a series of analyses, the analyst must interrupt the work, restart the system, and wait for a stable baseline and perhaps recalibrate. Since in-house gas generation allows for continuous operation, calibration of the detector simply requires the measurement of a standard sample at a user-specified interval to ensure that the system is operating properly.

A major benefit of in-house gas generators is that “once they are installed, you don’t have to worry about the gas supply,” according to Fran Diamond, a chromatographer at NMSLabs, a clinical and toxicology lab in Willow Grove, PA. This lab has used several Parker Hannifin hydrogen and nitrogen generators since 1996 and presently has ten units, installed into appropriate manifolds. Mr. Diamond reports that the maintenance requirements are minimal. They replace the filters and perform routine maintenance on the compressor for the nitrogen generators on a periodic basis and monitor the water in the hydrogen generators. John Kucowski, facilities manager at NMSLabs, reports that their four Tri-Gas generators have been operating very reliably and preventive maintenance is done about once a year. This maintenance can be conveniently performed on-site using Parker’s national third-party service provider, Mettler Toledo.

Minimizing the cost

An important advantage of an in-house generator is the economic benefit compared to the use of gas tanks or liquid nitrogen. The running cost of an in-house generator is extremely low, since the gas is obtained from compressed air or water and maintenance is a few hundred dollars per year for periodic filter replacement.

In contrast, when a gas tank is used, the actual cost is significantly greater than the cost of the tank. Dr. Ransom indicated that zero-air tanks are very expensive and the time and hidden costs including transportation, demurrage and paperwork when tank gas is employed impose a significant cost on the user. In addition, the time required transporting the tank, installing it, returning the used tank to storage and waiting for the system to re-equilibrate must be considered. While the calculation of the precise cost of the use of gas from tanks for a given user is dependent on a broad range of local parameters and the amount of gas that is used, significant potential savings can be obtained by the in-house generation of gas. A comparison of the cost of supplying gas via tanks versus the cost for use of an in-house gas generator is presented in Table 1. In this analysis, a tank of gas costing \$60 is consumed per week, and four are in-house (i.e., tanks are replaced monthly). In comparison, the maintenance cost of the in-house generator is for replacement of filters at perhaps \$1,000 per year, or approximately \$20 per week.

	IN-HOUSE GENERATOR	TANKS
Maintenance	\$800	\$0
Cylinders	\$0	\$3120
Demurrage	\$0	\$336
Labor (changing cylinders)	\$0	\$1040
Order processing	\$30	\$360
Shipping	\$50	\$3720
Invoice processing	\$10	\$120
Inventory control	\$0	\$72
Total	\$890	\$8768

Table 1. Annual costs: in-house generation vs. high-pressure tanks (in U.S. \$)

Conclusion

In-house generation of gases provides a safe, convenient and less costly method of providing the required gases. The gases are continually provided, and it is not necessary to replace tanks on a periodic basis. In addition to the cost reduction, less energy is required to supply gas, since tank gas or liquid nitrogen requires distillation of air (an energy-intensive process) and transportation of tanks to the final point of use.

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