

The Safe Way to Provide Gases for the Analytical Laboratory

Compressed gases such as zero air, nitrogen and hydrogen are commonly used in the laboratory for a broad range of instrumentation such as GC with FID, LC-MS and FTIR. In many laboratories, the compressed gas source is a high pressure gas cylinder. These cylinders are filled to a high pressure, typically, 2000 psi. The primary disadvantages of gas supply in the form of high pressure cylinders are safety, cost, and delivery and service inconveniences.

Precautions must be taken when transporting and handling high pressure compressed gas cylinders. If the individual loses control of the tank, it is possible that serious property damage and/or injury could occur. In the same vein, if a leak occurs, it is possible that the air in the laboratory could be displaced by the nitrogen or hydrogen, leading to asphyxiation. Users of high pressure compressed gas cylinders invariably will experience unplanned downtime resulting from an empty cylinder. This may occur in the middle of analysis, overnight, or during a weekend. Other inconveniences might include delayed delivery, inflexible delivery schedules, price increases, rental fees, and long term contracts.

These safety hazards and inconveniences can be eliminated by the use of an in-house gas generator. An in-house gas generator can be connected directly to the instrument and provides the necessary gas at the pressure and flow required by the application on a 24 hour/7 day a week basis with minimum user interaction. This paper describes how high purity gases can be generated in the laboratory and outlines the safety benefits that the generator provides.

The modern analytical laboratory requires highly purified gases to support the various modes of instrumentation that are commonly employed. As examples, Nitrogen is used with gas chromatography with flame ionization detection (GC-FID), thermal analysis (TA), Evaporative Light Scattering Detection (ELSD), Inductively Coupled Plasma Spectrometers (ICP), Liquid Chromatography with Mass Spectrometry (LC-MS) and Fourier transform infrared spectroscopy (FT-IR), Hydrogen is used as a fuel and a carrier gas for GC-FID while Zero Air is used for GC-FID, LC-MS and other techniques that are used to analyze for aromatic hydrocarbons.

Purified nitrogen and Zero air can be readily obtained from laboratory air and hydrogen can be generated from water in the laboratory using an in-house generator on a 24 hr/7 day basis.

An in-house generator provides a considerably greater level of safety, convenience, and cost savings than the use of high pressure compressed gas cylinders. In-house generators typically operate at low pressures and store small volumes of pressurized gas. This stored volume may vary from less than 50 cm³ to several gallons as compared to over 200ft³ of gas stored in high pressure gas cylinders. An in-house gas generator eliminates the need to handle heavy gas cylinders which has the potential risk of injury or damage caused by lifting, dropping, asphyxiation, and potential explosion. Gas generators provide convenience through the elimination of reliance on an external delivery service. Once the in-house gas generator has been installed, delivery of the desired gas is automatic, reliable, and relatively inexpensive. The cost of purchasing and operating a gas generator is attractive as compared to the use of high pressure cylinders. Paybacks are typically calculated at less than one year depending on the specific usage and required purity. Most importantly, the cost to operate and maintain an in-house gas generator is very low, especially relative to the cost of ordering, storing, and changing high pressure gas cylinders.

In-house generators are available for each gas, as well as generators for multiple gases to support the desired system; as an example a tri-gas generator is available which is commonly used to provide nitrogen, zero air and source exhaust air from laboratory air for use with LC-MS systems. In this paper, we describe how in-house gas generators operate and describe the safety benefits they provide.

In-house Generation of Nitrogen

Nitrogen can be obtained from compressed air in the laboratory using membranes (see Figure 1) that allow oxygen and water vapor to permeate the membrane while allowing nitrogen to flow through the membrane to the end use point. A typical system based on

this technology is the Parker Balston N2-45 Nitrogen Generator, (Parker Balston, Haverhill MA) which is recommended by all major LC/MS manufacturers. This system can produce up to 99.5 % pure nitrogen at a flow rate of 67 L/min at a maximum pressure of 145 psig. Dust and particulate matter are removed by a pre-filtration system and an oxygen monitor with an audible alarm is provided to signal a high oxygen concentration. If a high flow of nitrogen is required (e.g. to supply several GC-FID systems, pressure swing adsorption (PSA) systems which uses molecular sieves (activated carbon) can be used (Figure 2). The molecular sieve is extremely porous and can retain a significant amount of the pressurized nitrogen, which is then released by reducing the pressure. Flow rates of 99.9% Nitrogen as high as 470 SCFH can be obtained with the Parker Balston AGS400 generator.

In-house Generation of Hydrogen

In-house generation of Hydrogen gas is based on the electrolysis of water using either a metallic electrode or an ionomeric proton exchange membrane. When metallic electrodes are employed, a strong, water soluble electrolyte such as 20 % NaOH and a cathode in the form of a bundle of palladium tubes are used. Only hydrogen (and its isotopes) passes through the cathode while oxygen and other impurities collect at the anode, so that up to 1200 cc/min of hydrogen gas with purity in excess of 99.99999+% can be obtained (ParkerH21200NA). Alternatively a Proton Exchange Membrane (an ionic polymer membrane that is designed to conduct protons while being impermeable to gases such as hydrogen and oxygen can be employed). PEM's are commonly used in fuel cells to create an electric current (and form water) from hydrogen gas and oxygen gas. When an appropriate potential is applied to a PEM in the presence of water, the reverse process occurs and the water will be dissociated to form oxygen and hydrogen.

In-house Generation of Ultra Dry Air, Purge Gas, Zero Air and Source Exhaust Air Moisture, particulate matter and hydrocarbons can be removed from air using a coalescing filter which is a matrix of borosilicate glass hollow fibers in a fluorocarbon resin binder. For instrument grade air, two stage filtration is used which can remove 99.99 % of 0.01 μm particles and droplets and a three stage filter system is normally used to remove compressor oil vapor. A heated catalysis module and high capacity carbon absorption modules can be used in conjunction with coalescing filters to remove very light methane-based hydrocarbons.

Zero air can be generated by passing compressed air through pre-filters which serve to remove oil, water, dust and particulate matter. Compressor pump oil vapor and other hydrocarbons that may be present in the air are removed by then passing the filtered air through a heated catalytic convertor to form CO_2 and H_2O . The compressed air is then

passed through another particulate filter to remove dust and other solids and delivered to the analyzer.

Safety Benefits of In-house Gas Generation

In-house generation of gases provides a significant number of safety related benefits, including:

- The amount of gas that is generated by an in-house system in a given time period is relatively small and is can be controlled by the user. As an example, the Parker Balston H2PEM-100 hydrogen generator for GC-FID provides a flow up to 100cc/min at a delivery pressure up to 100 psig. In contrast, when a standard compressed hydrogen tank is employed, a full tank has a pressure that is greater than 2000 psi. If a leak were to occur in the tank or the tubing connecting it to the instrument, a significant amount of gas would be rapidly expelled into the laboratory. This gas could displace the equivalent amount of air and could lead to the asphyxiation of laboratory personnel.

When compressed gas tanks are used, it is necessary to replace the tank on a periodic basis. This requires that the tank must be transported from a storage location to the point of use. A standard tank is quite heavy and if the individual loses control of the tank during transport, it is possible that the valve will be compromised and the tank could become a guided missile, injuring personnel and/or damaging the laboratory.

- An in-house generator ports the gas directly into the instrument or manifold and provides the desired gas on a 24 hr/7day basis with minimal user interaction. In contrast, when a compressed gas system is employed, the possibility exists that the tank may run out in the middle of a run and you could lose valuable data if the samples are not analyzed properly.
- In-house generators monitor the flow and or /pressure of the gas. If there is a significant increase or decrease in the flow or pressure, a signal can be sent to the instrument to stop analyzing samples. This safety feature eliminates the possibility that erroneous data (or no data) will be collected.
- Compressed gas tanks must be strapped to a wall or laboratory bench to ensure that are secure. If the tank mounting device fails, a potential hazard exists. This is especially hazardous situation in laboratories in seismically active areas.
- While nitrogen could also be provided by the evaporation of liquid nitrogen, this approach leads to potential hazards which are eliminated by the use of an in-house generator. The use of liquid nitrogen leads to the possibility of freeze burns as the temperature of liquid nitrogen is -195.8°C , and a potential leak in the system could lead to a significant amount of gas in the laboratory.

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Figure 1 Separation of Nitrogen from Oxygen and Water Vapor via a Hollow Fiber Membrane

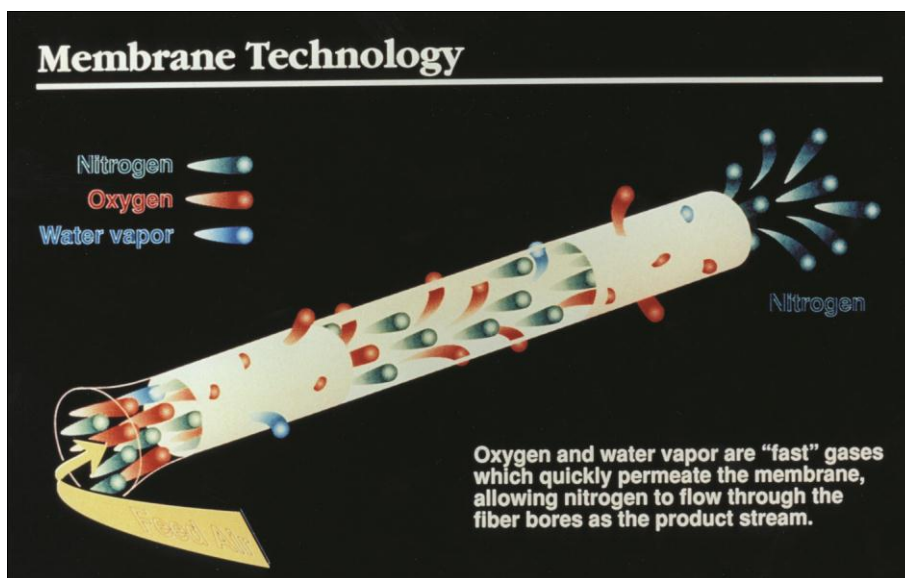


Figure 2 Generating Highly Purified Nitrogen via Pressure Swing Absorption

