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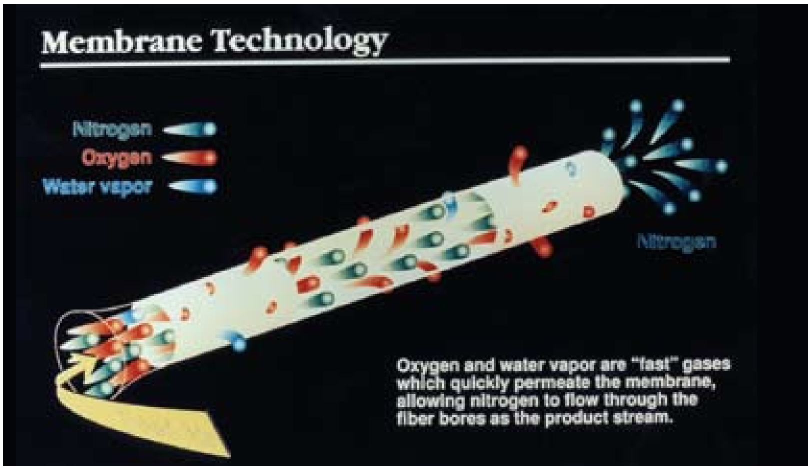
Selecting Makeup Gas for GC with FID

IN-HOUSE GENERATION PROMISES IMPROVED SAFETY, INCREASED CONVENIENCE AND REDUCED GAS COSTS ***by Phillip Allison and Peter Froehlich***

"The optimum flow rate for detection of the compounds of interest is typically significantly larger than the flow rate for the carrier gas."

When gas chromatography is employed for the detection of trace compounds, the characteristics of the carrier gas used for the actual separation and the gas used for detection may be significantly different. As an example, the carrier gas flow rate is selected to provide optimum resolution of the compounds of interest and is determined by the van Deempter relationship. The optimum carrier gas flow rate is a function of the compounds to be separated, the nature of the column, the temperature and a number of other considerations. Typically, the on-column carrier gas flow rate is in the order of a few mL/min. In contrast, the optimum flow rate for detection of the compounds of interest is typically significantly larger As an example, when a flame ionization detector (FID) is employed, the detector gas flow has to maintain a sufficiently high concentration of electrons for ionization of the compounds of interest and must be capable of sweeping the solute through the detector so that sharp peaks can be obtained for highly retained peaks. The flow rate for optimizing the detection is frequently as high as 500-2000 mL/min.

▼ *Figure 1: Separation of nitrogen from oxygen and water vapor via a hollow fiber membrane.*



In addition to flow rate differences, the chromatographer may be concerned with the composition of the carrier gas and the detector gas. While low levels of impurities such as hydrocarbons will likely have little effect on the actual separation, they could dramatically increase the background and/or the noise from the detector, thereby reducing the sensitivity of the analysis, and a higher level of purity may be required for the detector gas than for the carrier gas.

To increase the gas flow to what is required for the detection step, a "makeup" gas such as helium or nitrogen is delivered to the chromatographic system between the column and the detector. The makeup gas should be selected so that it does not affect the fuel and oxidant balance, and it must be fairly inert to the detector so that it does not affect the report of the concentration of the compound(s) of interest. In many facilities, the makeup gas is provided from a cylinder or tank. While this approach works, an in-house makeup gas generator can provide a higher level of purity than bottled helium or bottled nitrogen. In addition, an in-house makeup gas generator can provide a considerably safer, more convenient and less expensive approach to supply the required gas.

Generation of zero-grade nitrogen and zero-grade air using an in-house generator

Zero-grade nitrogen for makeup gas can be readily obtained from laboratory compressed air using an in-house generator (model MGG-400NA or MGG-2500NA FID MakeupGas generator, Parker Filtration and Separation Division, Haverhill, Massachusetts) that includes a heated catalytic converter that is similar to an automobile exhaust system. The converter includes a proprietary catalyst blend that is combined with platinum and a hollow fiber membrane separator.

The heated catalyst is used to remove all hydrocarbons by converting them to CO^2 and water vapor, while the hollow fiber membrane allows the separation of nitrogen from oxygen and water vapor. The hollow fiber membrane module (Figure 1), which is the heart of the system, is designed to preferentially allow oxygen and water vapor in the air to quickly permeate the membrane wall while nitrogen travels through the hollow fiber out the end. A schematic diagram of a typical system for the generation of nitrogen is shown in Figure 2.

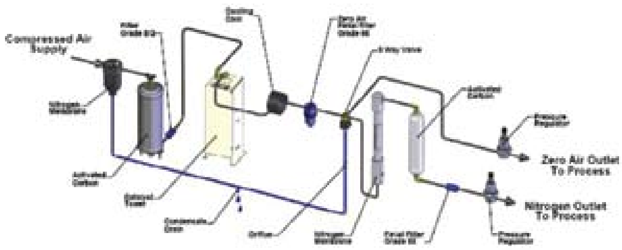


Figure 2 : Schematic diagram of FID makeup gas generator.

The hollow fiber used to separate the nitrogen has a small internal diameter, and thousands of fibers are bundled together to provide a large surface area for the desired flow of nitrogen, as shown in Figure 3. The makeup gas generator can produce makeup nitrogen with purity of better than 99.9999 percent with respect to hydrocarbons (< 1 ppm) at a maximum flow rate of 400 mL/min. In addition, the purity of the nitrogen is greater than 99 percent with respect to oxygen. In addition, the in-house generator can produce zero-grade air with a hydrocarbon concentration that is less than 0.05 ppm at flow rates up to 2500 mL/min. Figure 4 shows a comparison of zero-grade air that was produced by the MakeupGas generator and zero-grade air that was obtained from bottled fuel air from a commercial supplier. It shows that the gas generated by the generator is purer than that from bottled fuel air, as it provides an extremely flat baseline with essentially no signal due to hydrocarbons, while the zero-grade bottled air provided an irregular baseline with a significant level of hydrocarbons, which could impact analysis. Dr. Nithy Govindarajah, a scientist at Symrise Laboratories in Branchburg, New Jersey, reports that he has used the MakeupGas generator with three GCs and analyzed over 1000 samples of essential oils a month, and always obtained a flat baseline.

Figure 3: A bundle of hollow fibers provides a high flow of zero-grade nitrogen.

Minimizing safety hazards

When a makeup gas generator is employed, only a small amount of the gas is present at a low pressure at a given time and the gas is ported directly to the GC. The system generates a maximum of 2.5 L/min of air or 400 mL of nitrogen at a maximum pressure of 120 psig of nitrogen. If a nitrogen leak were to occur with the generator, there would be a negligible change in the composition of the laboratory air, with only trace nitrogen dissipating harmlessly.

In contrast, a number of serious hazards exist when makeup gas is supplied to the GC via a tank. As an example, if the contents of a full tank of helium or nitrogen were suddenly vented into the laboratory, up to 9000 L of gas would be released. This volume would displace the laboratory air, reducing the breathable oxygen content and potentially creating an asphyxiation hazard for the laboratory occupants. Another potential hazard that is eliminated by use of a makeup gas generator is injury or damage while transporting and installing a gas tank. A standard tank is quite heavy and can become a guided missile if the valve on a full tank is compromised during transport (in many facilities, specially trained technicians are used to replace gas tanks).

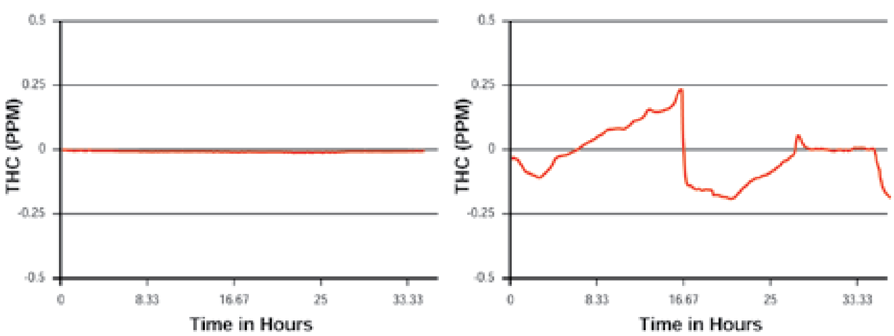


Figure 4: FID baseline from Makeup Gas generator (top) and from bottled fuel air (bottom). Flow rate = 450 mL/min.

Convenience

When an in-house generator is employed, the gas is supplied on a continuous basis and can be provided on a 24-hour, 7-day-a-week basis without any user interaction other than a minimum of routine annual maintenance. In contrast, when tank gas is employed, the user must pay close attention to the level of gas in the tank and replace the tank on a periodic basis. The in-house system obviates the need to obtain a replacement tank. In many facilities, spare gas tanks are stored outside in a remote area for safety reasons, and it is time-consuming to get a replacement cylinder. When it is necessary to get a replacement makeup gas tank, the chromatographer may require an individual who is qualified to handle the tanks. Many users have indicated that replacing used tanks can be a significant inconvenience, especially in inclement weather if the tanks are stored outside or if not properly secured when the laboratory is located in a seismic zone.

If the need for replacement occurs during a series of analyses, the analyst must interrupt the analytical work to restart the system and wait for a stable baseline, and may have to recalibrate the system. In addition, if a series of automated analyses is desired (e.g., overnight or over a weekend), the analyst must ensure that a sufficient volume of each gas is on hand before starting the sequence.

The frequency of tank replacement depends on the usage of the system. Changing the tank is clearly an inconvenience and leads to a reduction in the useful operating efficiency of the facility. In addition to the actual time required for changing the tank, the laboratory staff must verify that there are sufficient replacement tanks in storage and order replacement tanks as appropriate. The use of a makeup gas generator eliminates the need to keep track of and change gas cylinders. Dr. Govindarajah indicated that he previously had to replace the gas tank approximately three times a month when tank gas was used for makeup gas, and now simply turns on the generator, saving time and eliminating inconvenience. Similarly, Dr. Mike Jordan of Agriculture Canada (Kentville, Nova Scotia, Canada), who analyzes volatile anaerobic compounds in fruits, indicated that the generator allows him to leave the FID detectors on the gas chromatographs powered up on a 24/7 basis. This saves considerable time and increases laboratory efficiency, as it is not necessary to calibrate the detector every time it is turned on. Dr. Jordan simply runs a standard sample on a periodic basis, which takes only a few minutes, to ensure that the system is operating properly. An additional benefit is that it is no longer necessary to train each technician in the calibration process.

Cost

In addition to safety and convenience, another benefit of a makeup gas generator is the cost compared to the use of gas tanks. The cost of operation of the generator is extremely low, as the raw materials to prepare the required gas are air and electricity. Running costs and maintenance for the generator add up to a few hundred dollars a year.

In contrast, the cost for using makeup gas from tanks includes the actual cost of obtaining the gas tank as well as the time involved in changing tanks, ordering new tanks, maintaining inventory and related activities. While calculating the precise cost of using makeup gas from tanks for a given user is dependent on a broad range of local parameters and the amount of gas that is used, we present below an overview of the potential savings from the use of an in-house makeup gas generator.

It should be noted that there are many hidden costs, including transportation costs, demurrage costs and the required paperwork (e.g., a purchase order, inventory control and invoice payment) when tank gas is employed. In addition, the time that is required to transport the tank from the storage area, install the tank, replace the used tank in storage and wait for the system to re-equilibrate represents money as well.

A comparison of the cost of supplying gases via tanks versus a makeup gas generator is presented in Table 1. For this analysis, we assumed that a single tank of makeup gas is consumed each week by each chromatograph and that the cost of each tank is $60 (this approximation ignores the incidental costs of handling the gas tank, downtime, ordering tanks, etc.). In comparison, the cost of using the generator is approximately $50 per week. Since the cost of supplying makeup gas is significantly lower with the generator than with tank gas, it is now possible to leave the FID detector on continuously.

Table 1.	In-House Generator	
Electrical Power	$380	$0
Maintenance (compressor)	$1,500	$0
Maintenance (generator)	$800	$0
Cylinders	$0	$3,120
Demurrage	$0	$840
Labor (changing cylinders)	$0	$1560
Order Processing	$30	$360
Shipping	$100	$3,720
Invoice Processing	$10	$120
Inventory Control	$0	$72
Other	$2,820	$9792

Assumptions:
52 cylinders at $60/cylinder
10 cylinders in use (5 in use, 5 filled) at $7/mo
$30 labor/cylinder
1 order/month @ $30 processing costs
$20/cylinder

Table 1 Annual costs, in-house generation versus high pressure tanks (in U.S. $)

Conclusions

An in-house makeup gas generator can provide high-purity nitrogen and zero-grade air at the flow rates required for the use of flame ionization detectors in gas chromatography. The hydrocarbon content of the makeup gas generated is considerably lower than that in bottled gas from external sources. In addition to the higher purity of the makeup gas, an in-house generator is safer, more convenient and less costly than bottled gas. These benefits allow the chromatographer to maintain power to the detector on a continuous basis, obviating the need for frequent, time-consuming recalibration.

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