

# Whatman Gas Generators

Restek Purus™ Gas Systems in alliance with Whatman



Chromalytic Tech Fax : +61 3 9761 1169

## Features & Benefits

- Eliminate costly, inconvenient gas cylinders from the laboratory
- Gas Supply Systems available for FT-IR, GC-FID, NMR, TOC, and other laboratory instruments
- Improve instrument accuracy, sensitivity, and performance
- Safe - operate at low pressures
- Cost effective - payback typically less than one year
- Compact - free up valuable laboratory floor space
- Recommended by most instrument manufacturers

### Gas Generator Selection Chart

Model #	Flow Capacity
<b>Hydrogen Generators</b>	
75-30	80 cc/min.
75-32	150 cc/min
75-34	300 cc/min
75-36	550 c/lmin
<b>Nitrogen Generators</b>	
75-72	44 lpm
75-720 wil 0.2 monitor	44 lpm
75-880	70 lpm
76-92	1100 cc/min
76-94	1100 cc/min
76-96 (2)	2000 cc/min
<b>Zero Air Generators</b>	
75-83	1 lpm
76-803	3.5 lpm
76-807	7 lpm
76-818	18 lpm
76-830	30 lpm
<b>Gas Generator for TOCs</b>	
78-40	1250 scclmin
<b>FT-IR Purge Gas Generators</b>	
75-45 (1)	14 lpm
75-52 (1)	28 lpm
75-62 (1)	85 lpm
74-5041(3)	28 lpm
<b>Compressed Air Dryers</b>	
75-05 (1)	12 lpm
75-10 (1)	57 lpm
64-20 (1)	283 lpm
<b>Membrane Air Dryers</b>	
64-01	71 lpm
64-02	142 lpm
64-10	708 lpm

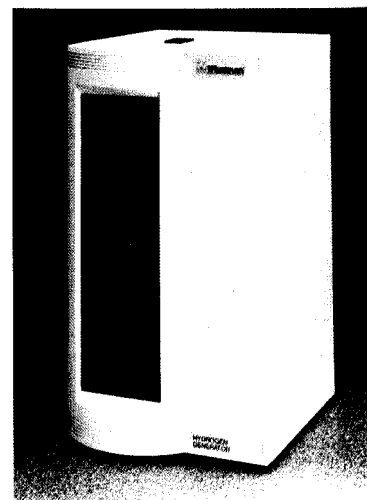
- (1) House compressed air supply of at least 60 psig or more is required  
 (2) House compressed air supply of at least 75 psig or more is required  
 (3) Includes air compressor

Whatman Gas Generators completely eliminate dangerous, costly, inconvenient gas cylinders from the laboratory. There is no longer any need to buy and store excessive reserves, and use up valuable laboratory floor space to protect yourself from late deliveries, transportation interruptions, or periods of tight supplies. With a Whatman Gas Generator, you control your supply! Payback periods are typically less than one year. Whatman Gas Generators produce an unlimited supply of consistently pure gas eliminating downtime due to "bad gas" or empty cylinders. The time-consuming task of recalibrating instruments is also eliminated.

### Applications

- Produce 99.99999% pure hydrogen for GC-FIDs
- Provide zero air to GC-FIDs
- Generate ultra high purity nitrogen for laboratory applications
- Provide clean, dry instrument air at -100°F dew point
- Provide clean, dry, CO2 free air to FT-IR Spectrometers
- & Provide a carrier/combustion gas for TOC analyzers

Quality products by Whatman Inc. are made in the U.S.A., and each one incorporates superior workmanship and the best quality components available. All products are backed by the Restek field sales and customer service team and a one year warranty. Your complete satisfaction is guaranteed.



A Whatman Hydrogen Generator



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# RESTEK

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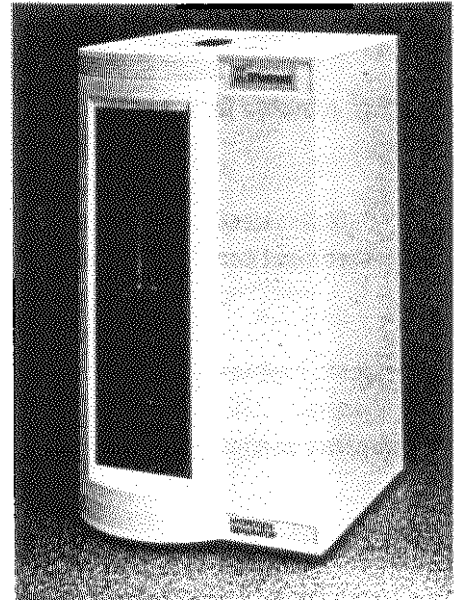
# Whatman Hydrogen Generators

(Flow Capacity of up to 550 cc/min)



## Restek Gas Systems in alliance with Whatman

- **Eliminate dangerous and expensive hydrogen gas cylinders from the laboratory**
- **Safe - produces only as much gas as you need**
- **Produce a continuous supply of 99.99999% pure hydrogen gas, ideal for carrier and fuel gas applications**
- **Compact and reliable-only one square foot of bench space required and designed to run continuously 24 hours/day**
- **LED displays indicate system status at a glance**
- **Easy annual maintenance**
- **Certified for laboratory use by CSA, UL, IEC 1010, and CE**



**A Whatman  
Hydrogen  
Generator**

The **Whatman** Models 75-30, 75-32, 75-34, and 75-36 Hydrogen Generators eliminate the need for expensive, dangerous, high pressure cylinders of hydrogen in the laboratory. It is no longer necessary to interrupt important **analyses to change cylinders**. Generator flow capacities of up to 550 cc/min. of ultra-high purity hydrogen are available.

The **Whatman Hydrogen Generators** are compact **benchtop** units designed for use in the **laboratory** or in the field. **Hydrogen gas is produced by electrolytic dissociation of water**. The **resultant hydrogen stream then passes**

through a palladium membrane. Only hydrogen and its isotopes can penetrate the palladium membrane; therefore, the purity of the output gas is guaranteed to be 99.99999+% consistently. This technology produces hydrogen at a purity two **orders of magnitude** greater than competitive technologies. **Whatman Hydrogen Generators offer many special features to ensure safe and convenient operation**. These features include low-water audible alarms to indicate when the water reservoir needs filling and automatic shutdown to protect extensive laboratory equipment.

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**RESTEK**

# Whatman Nitrogen Generation Systems

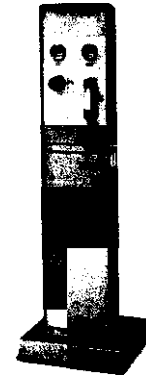
Models 75-72, 75-720, 75-880



Restek Purus Gas Systems in alliance with Whatman

- Eliminates the need for costly, dangerous, inconvenient nitrogen cylinders in the lab
- Model 75-72 requires no electricity
- Compact design frees up valuable lab floor space
- Models 75-720 and 75-880 include Oxygen Analyzers
- Offers long-term cost stability-uncontrollable vendor price increases, contract negotiations, long-term commitments, and tank rentals are no longer a concern

Whatman  
Nitrogen



75-72 Nitrogen  
Generator

The Whatman 75-72, 75-720, and 75-880 Nitrogen Generation Systems produce up to 1.50 SCFH of compressed nitrogen, on-site. The purity level of the nitrogen stream is defined by the user, for the application, and may range from 95% to 99.5%

The Model 75720 Nitrogen Generator includes an oxygen analyzer which monitors the oxygen concentration of the nitrogen stream. An audible alarm signals high or low oxygen concentrations

The Model 78-880 Nitrogen Generator also includes an oxygen analyzer and is designed for higher flow and purity applications. This system is ideal for large volume turbo solvent evaporators.

Whatman Nitrogen Generators are complete systems engineered to transform standard compressed air into nitrogen at safe, regulated pressures, on a continuous basis, without the need for operator attention. The systems eliminate the need for costly, dangerous dewars and cylinders in the lab. Nitrogen is produced by utilizing a combination of filtration and membrane separation technologies. A high efficiency prefiltration system pretreats the compressed air to remove all contaminants down to 0.1 micron. Hollow fiber membranes subsequently separate the clean air into a concentrated nitrogen output stream and an

oxygen enriched permeate stream, which is vented from the system. The combination of these technologies produces a continuous output flow of pure nitrogen. Typical applications include: chemical and solvent blanketing, glove box purging, chemical and solvent evaporation, instrument purge and supply, evaporative light scattering detector use (HPLC), and sparging.

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**RESTEK**

# Whatman Ultra High Purity Nitrogen Generator

Models 76-92, 76-94, 76-96 (Flow Capacity: 1.1 lpm to 2 lpm)



*Restek Purus Gas Systems in alliance with Whatman*

- Produces a continuous supply of high purity nitrogen gas from existing compressed air
- Eliminates the need for costly, dangerous, inconvenient nitrogen cylinders in the laboratory
- Compact design frees up valuable laboratory floor space
- Offers long term cost stability - uncontrollable vendor price increases, contract negotiations, long term commitments and tank rentals are no longer a concern
- Ideal for carrier gas or solvent evaporation applications



A Whatman Model  
7644 Ultra High  
Purity Nitrogen

The Whatman 76-92 and 76-94 UHP Nitrogen Generators can produce up to 1.1 lpm of ultra high purity nitrogen gas. These systems are completely engineered to transform standard compressed air into 99.9995% nitrogen, exceeding the specification of UHP cylinder gas. Nitrogen is produced by utilizing a combination of state-of-the-art purification technologies and high efficiency filtration. The 76-96 can produce up to 2 lpm of 99.99% pure nitrogen gas from standard compressed air.

Pressure swing adsorption is utilized for the removal of O<sub>2</sub>, CO<sub>2</sub>, and water

vapor. A catalyst module is incorporated in the 76-94 to oxidize hydrocarbons from the inlet air supply.\* The generators also have high efficiency coalescing prefilters and a 0.01 micron (absolute) membrane filter incorporated into their design. The Whatman UHP Nitrogen Generators are engineered and packaged in a small cabinet to fit nearly any benchtop. The systems eliminate the need for costly, inconvenient high pressure nitrogen cylinders. The 76-92 and 76-94 are ideal for carrier gas applications. The 76-96 is ideal for purging ICP's and can be used for solvent evaporation applications (on select equipment).

\*Model 76-92 has the same system specifications as Model 76-94 except for hydrocarbon removal.

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**RESTEK**

# Whatman Zero Air Generators

Models available to service up to 100 FIDs

*Restek Purus™ Gas Systems in alliance with Whatman*

**Produces UHP Zero Air from house compressed air**

**Eliminates inconvenient and dangerous zero air cylinders from the laboratory**

**Increases the accuracy of analysis and reduces the cleaning requirement of the detector**

**Recommended and used by many GC and column manufacturers**

**Payback period of typically less than 1 year**

**Silent operation and minimal operator attention required**

**Models available to service up to 100 FIDS**

## Convenient

**Whatman Zero Air Generators** are complete systems with state-of-the-art, highly reliable components **engineered for easy installation**, operation, and long term performance. **Whatman Zero Air Generators** are much easier to install than dangerous, high pressure gas cylinders, and only need to be installed once! All that is required is a **standard compressed air line and an electrical outlet.**

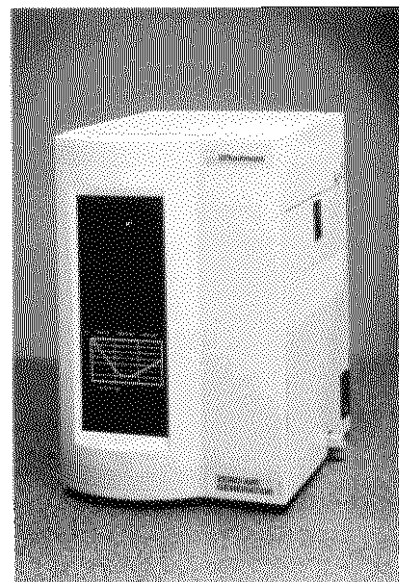
## Easy to Operate

**Whatman Zero Air Generators** are easy to operate, there is no complicated operating procedure to learn or any labor intensive monitoring required.

## Cost Effective

**Whatman Zero Air Generators eliminate all the inconveniences and costs of cylinder gas supplies and dependence on outside vendors.** Uncontrollable vendor price increases, contract negotiations, long term commitments and tank rentals are no longer a **concern; Whatman Zero Air Generators offer long term cost stability.**

**There is no need to use valuable laboratory floor space to buy and store excessive reserves to protect yourself from late deliveries, transportation interruptions, or periods of tight supplies.** With a **Whatman Zero Air Generator**, you control your



**Models 76-803,  
76-807, 76-818,  
76-830**



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# **American Laboratory**

The effect of detector gas  
purity on FID baseline  
stability

By Eugene Barry

## The shift to on-site gas generation

By Robert Daly

**O**VER THE PAST three decades we have experienced many changes in our culture: in our homes, governments, institutions, and occupations. Technological advancement has resulted in the elimination of time-honored traditions and behaviors. In many instances, we have forever changed the way we do things. This phenomena is often described as a paradigm shift. The word paradigm is derived from the Greek word *paradeigma*, which means pattern, example, or model. A paradigm shift is the change from a traditional way of acting, thinking, or doing, to newer, more effective ways.

Paradigm shifts are often motivated by the availability of new technologies and typically result in obvious benefits such as increased efficiency and convenience, improved performance, reduced cost, and an ability to solve difficult problems. Two examples of a paradigm shift are elimination of the home delivery of ice by the invention and commercialization of the refrigerator/freezer and replacement of the home delivery of milk in reusable glass bottles by disposable packaging. More recently, the delivery of water deionization tanks to laboratories has been replaced by the availability of point-of-use water purification systems; personal computers have resulted in internal corporate desktop publishing, and a paradigm shift in Japanese manufacturing has resulted in the changed perception of Japanese products from poor quality during the 1960s and

1970s to among the highest quality in the world during the 1980s and 1990s. It is easy to imagine the future obsolescence of video rental stores resulting from the availability of movies through a fiber optic superhighway and replacement of the current internal combustion engine by one that is powered by alternative fuels such as natural gas, electricity, or hydrogen.

Many such paradigm shifts have occurred in the laboratory, including the use of autosamplers, computer-controlled instrumentation, automated titration, fused-silica capillary columns, and the use of supercritical fluids for extraction.' This article describes the paradigm shift occurring within the laboratory related to the method for supplying compressed gases used for a variety of purposes.

### *Applications for compressed gases*

Many applications exist for compressed gases in the laboratory. These include purge gas, carrier gas, and fuel gas for instruments such as Fourier transform infrared spectrometers, gas chromatographs, total organic carbon analyzers, nuclear magnetic resonance spectrometers, and thermal analyzers. Noninstrument applications include solvent evaporation, purging of laser gas chambers, use with autosamplers, and blanketing of solvents and samples. The gases typically used for these applications include air, nitrogen, hydrogen, helium, argon, and various mixtures. The required gas purity depends on the application and can range from filtered air to research-grade nitrogen with less than 1 ppm contaminant concentration.'

For these applications, compressed gases are an important utility. Much like electricity, water, natural gas, heat, and telephones, compressed gases are essential to the operation of the laboratory. Consequently, the source of this utility should be convenient, reliable, safe, and cost effective. As with many other utilities, the supply of compressed gases should be simple and taken for granted. Currently, the availability of on-site, point-of-use gas generators is allowing scientists and managers to treat the supply of compressed gases as a utility as opposed to a contracted service.

### *Traditional source of compressed gases*

Ever since compressed gases have been used in the laboratory, the accepted source has been the high-pressure gas cylinder. These cylinders are filled to a high pressure, typically 2200 psig, with the required gas by a supplier operating a gas production facility. The cylinders are delivered to customers upon purchase and picked up when empty. The empty cylinders are then refilled and delivered to another customer.

The primary disadvantages of gas supply in the form of high-pressure cylinders are delivery and service inconveniences, safety, purity, and cost (including hidden cost). The advantage of high-pressure cylinders is the availability of a wide range of gases for applications in a laboratory.

Several types of inconvenience are frequently experienced in the use of delivered high-pressure gas cylinders. Every user of gas cylinders will eventually experience unplanned downtime as a result of an empty cylinder. This may occur

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# American Laboratory

NEWS EDITION

## **Benchtop Gas Generators Increase Instrument Accuracy and Reduce Laboratory Operating Costs**

**THE EFFECT OF FUEL AIR PURITY ON FID  
SENSITIVITY**

By Dorothea J. Jeffery, Gregory C. Slack,  
and Or. Harold M. McNair

**ULTRA PURE HYDROGEN AS A CARRIER GAS FOR  
CAPILLARY CHROMATOGRAPHY**

By Dr. Eugene Barry

# Benchtop Gas Generators Increase Instrument Accuracy and Reduce Laboratory Operating Costs

## THE EFFECT OF FUEL AIR PURITY ON FID SENSITIVITY

By Dorothea J. Jeffery, Gregory C. Slack, and Dr. Harold M. McNair

In the field of capillary gas chromatography, the presence of sensitive detectors and trace analyte samples increases the need for dry, clean fuel gases. Laboratories in an industrial setting often maintain several gas chromatographs in continuous operation. Since large volumes of fuel gases are consumed daily, gas cylinders are changed almost as frequently. Usually the fuel air is of breathing quality and is introduced either directly or after drying via a molecular sieve trap. The objective of this study is to compare flame ionization detector sensitivity vs. air purity under isothermal conditions. This study included air sources as follows: the Whatman@ Zero Air Generator, breathing air (cylinder without scrubbers), ultra zero air (cylinder), and filtered house air.

The study proceeded as follows:

- 1 compared baseline runs taken at 10 minutes, 60 minutes, and 12 hours
- 2 compared runs of a 50 ppm trace alkane sample, and
- 3 compared runs of a 1 ppm trace alkane sample for the air sources with the exception of the house air. Finally, both breathing and generated air studies were repeated under optimized conditions and without air scrubbers. This final study also included the filtered house air.

A comparison of the chromatograms for the baseline and the trace component runs showed that both the Whatman Zero Air Generator and the ultra pure air produced lower signals and better sensitivity: as shown by increased peak area counts. These baselines were also more stable than either the breathing air or the house air. In addition to the lower and stable baseline, the air generator had the advantage of providing a continuous source of air.

### Results

Optimized carrier gas flow rate and split ratio were used in order to produce better quantitation. Similar to previous baseline runs, the zero air signal (average signal during blank runs) was lower than either of the other air sources (Figure 1 and Table 2). In addition, the zero air undecane peak area counts for both the 50 ppm and 1 ppm standards were significantly larger than those of either the breathing or house air (Table 2). Tables 3 and 4 contain the statistical comparisons of the average peak area counts for both the 50 ppm and 1 ppm standards respectively.

Whatman zero air was used as the reference in the paired t value and 2-tail probability determinations. The paired t value critical values at the 97.5 confidence level were 3.18 for 3 degrees of freedom and 4.30 for 2 degrees of freedom. The calculated values in both the 50 ppm and the 1 ppm runs were larger than the respective critical values; therefore, the differences in area counts were not due to random fluctuations. In addition, the 2-tail probabilities were below the absolute critical value of 0.05. This occurrence supports the theory of non random differences in area counts as determined by the paired t test. Since both the paired t values and 2-tail probability values were outside their respective critical ranges, the differences in peak areas were not due to random fluctuations'. These differences were due to the flame purity.

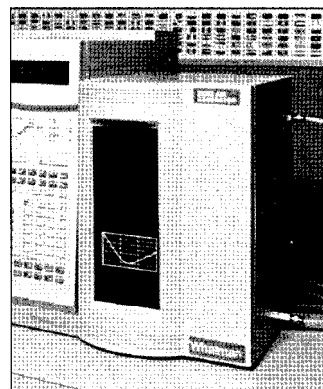


Exhibit A: A Whatman Zero Air Generator

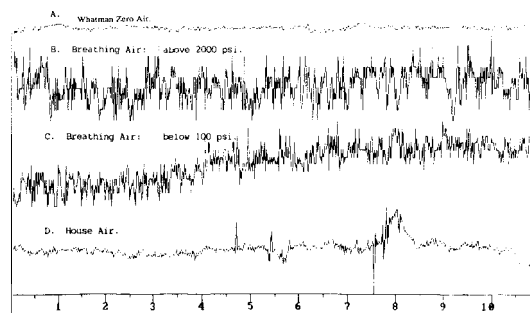


Figure 1: Baseline Signals - Random spike at 6 min. for Zero Air (A). Baselines are raw data (mA) on equivalent scales.

**Table 2: Baseline Data**

	Air Source			
	Whatman	Breathing Air >2000 psi	Breathing Air <100 psi	House Air
Signal	12	22	10	22

**Table 3: Undecane Peak Area Data - 50 ppm Standard Data**

Mean Area Counts	10878	9784	9181	8206
Standard Deviation	426	194	179	89
Paired t Values	-	3.8	a.75	14.82
Probability (Z-Tailed)	-	0.032	0.003	0.001
Degrees of Freedom	3	3	3	3

**Table 4: Undecane Peak Area Data - 1 ppm Standard Data**

Mean Area Counts	1744	1629	1531	1404
Standard Deviation	2.1	35.2	20.8	21.1
Paired t Values	-	4.77	19.57	27.06
Probability (Z-Tailed)	-	0.041	0.003	0.001
Degrees of Freedom	2	2	2	2

### Conclusion

The Whatman Zero Air Generator has advantages over the conventional sources of air for GC analysis. A lower and more stable baseline signal can be obtained. Due to lower baseline noise, the signal-to-noise ratio is larger, giving rise to higher sensitivity or larger peak areas. A comparison of peak areas for the alkane standards gave similar results. The air generator produced peak areas which were more than 12% of the breathing air peak areas. Not only does the air generator give better baselines, it also eliminates the need for frequent cylinder changes, thus saving time.

<sup>1</sup> Abacus Concepts, Statview II, Abacus Concepts, Inc.: California 1987.

*Dr. Harold M. McNair is Chairperson of the Chemistry Department at Virginia Polytechnic institute and State University. Dorothea J. Jeffety and Gregory C. Slack are currently undergraduate students at VPI & SU.*

## ULTRA PURE HYDROGEN AS A CARRIER GAS FOR CAPILLARY CHROMATOGRAPHY

By Dr. Eugene Barry

Traditionally, chromatographers have used helium and nitrogen as the carrier gases of choice in gas chromatography. Now, with the availability of a reliable, safe, ultra pure Whatman Hydrogen Generator from Balston Inc. (Haverhill, MA) for the laboratory, the use of hydrogen as a carrier gas for capillary G.C. has advantages worth considering.

A comparison of the Van Deemter curves for nitrogen, helium and hydrogen is illustrated in Figure 1.

The small slope after the optimum flow velocity would mean that flow velocities could be increased without too much loss in efficiency (increase in H). Increased flow velocities will shorten analysis time (often cutting analysis time in half), resulting in lower elution temperature requirements, lower cost per analysis, and extended column life. From a theoretical yet practical viewpoint, the use of Hydrogen as a carrier gas allows the generation of nearly four times as many effective theoretical plates per second as Nitrogen. From a practical chromatographer's view, the chance of picking a good flow rate with the first experiment is increased.

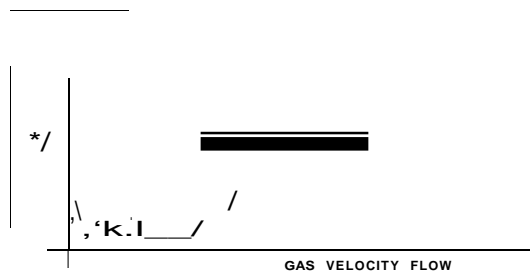


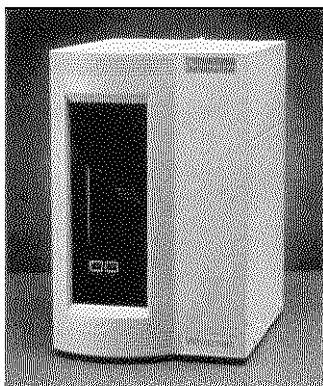
Figure 1: A Comparison of the Van Deemter Curves for Nitrogen, Helium, and Hydrogen.

### Summary

Very acceptable separations and increased sensitivity for trace analyses can be obtained by using hydrogen generated by the Whatman Hydrogen Generator as a carrier gas. This unit provides a safe, reliable, economical source of very pure hydrogen. Cost figures show that the 300 ml/min Whatman generator pays for itself in 1 to 2 years. The hydrogen is produced by electrolysis through palladium, which produces ultra-dry hydrogen with less than 10 ppb impurities. Only 50 ml of hydrogen are stored at any time in the unit, ensuring complete safety and compliance with OSHA and NFDA regulations.

With this new, safe source of ultra pure hydrogen available, chromatographers are experiencing some additional benefits with the use of hydrogen as a carrier gas. These benefits would include the apparent ability of hydrogen to repel or counteract trace amounts of oxygen (as opposed to nitrogen and helium which accumulate trace amounts of oxygen). The columns last longer because they are subjected to shorter runs of cooler temperatures, typically increasing their useful life by 33%.

"The predominant opinion-that the gas with the lower viscosity, that is, hydrogen, is the best carrier gas..." for capillary G.C. has been expressed by Rohrschneider and Pelster. The Whatman Ultra High Purity Hydrogen Generators are the only Generators available that enable Chromatographers to use hydrogen for both carrier gas and fuel gas applications. Hydrogen is being put into use in more laboratories with the availability of a safe, economical, ultra pure Whatman hydrogen generator from Whatman Inc.



**Exhibit B:** A Whatman Hydrogen Generator

*Dr. Eugene Barry is a Professor of Chemistry and Graduate Coordinator at the University of Massachusetts, located in Lowell.*

#### FOR ADDITIONAL INFORMATION

Whatman Inc., also manufactures and markets a complete line of Whatman Gas Generators including: Ultra High Purity Nitrogen Generators, Gas Generators for FT-IR Spectrometers and NMR's, TOC Gas Generators, and Complete Systems with Oil-less Compressors. All generators are designed to enhance instrument accuracy and performance, and increase laboratory efficiencies by automating gas delivery systems. Detailed information is available by contacting Whatman Inc., 260 Neck Road, Haverhill, MA 01835. Call toll-free at 1-800-343-4048, or fax 1-978-374-7070.

# Whatman®

Whatman Inc.  
260 Neck Rd., Box 8223  
Haverhill, MA 01835-0723  
800-343-4048 or 978-374-7400  
Fax: 978-374-7070

Whatman Canada Ltd  
2495 Haines Rd.  
Mississauga  
Ontario L4Y 1Y7  
905-272-1516



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ity on-site generators is typically very consistent and dependent upon adherence to maintenance schedules. On-site generators deliver gases through the same flow system from the day they are installed until they are decommissioned. Thus, the gas purity is not influenced by a change in the materials of contact, as it is with high-pressure cylinders.

Gas generators provide increased safety in comparison to high-pressure cylinders. The generators typically operate at low pressures (such as 100 psig) and store small volumes of pressurized gas. This stored volume may vary from less than 50 cm<sup>3</sup> to several gallons as compared to over 200 ft<sup>3</sup> of gas stored in high-pressure gas cylinders. Gas generators eliminate the need to handle heavy gas cylinders, which is a risk of injury or damage caused by lifting, dropping, asphyxiation, and potential explosion. Safety issues with gas generators concern the use of electrical and mechanical components. These types of concerns should be relieved if the generators are designed and tested to Underwriters Laboratories (UL), Canadian Standard Association (CSA), and International Electrotechnical Commission (IEC) specifications.

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. Perhaps most importantly, the cost to operate and maintain a gas generator is very low, especially relative to the cost of ordering, storing, and changing high-pressure gas cylinders. Table 2 shows a cost analysis for a typical installation of a gas generator to replace the use of high-pressure gas cylinders. This cost analysis of a hypothetical laboratory replacing high-pressure gas cylinders with an on-site gas generator shows a payback of less than six months.

Disadvantages to on-site generation should also be mentioned. Generation equipment may need to be budgeted for purchase as opposed to paying for delivered gas through an expense account. Users of gas generators should plan for present and future gas require-

Table 2

Ultrazero-grade air cost comparison: On-site generator versus high-pressure cylinders'

cost component	Gas generator	High-pressure cylinder
Gas generator price	\$2885	NA
Annual dower cost	\$15	NA
Annual maintenance cost	\$411	NA
Annual cylinder gas price	NA	\$5700
Annual cylinder demurrage	NA	\$126
Annual labor cost to change cylinders	NA	\$475
Annual order processing cost	\$30	\$360
Annual shipping cost	\$10	\$570
Invoice payment cost	\$10	5120
Inventoly Control cost	NA	\$40
TCdCd	\$3361	57391

\*Gas genemlorcost is based on a model 76.803NA (Whatman Inc., Haverbill, MA), which requires 100 VA power.

Cylinder gas cost is based on 3 slpm flow rate of ultrazerograde air for 8 k/day, 260 day/yr, at a price of \$100/cylinder.

Other cylinder costs include demurrage at \$5.25/cylinder per month, cylinder change labor at 10 min/cylinder and \$50 hr, order processing cost of \$10/order, shipping cost of \$10/cylinder, invoice payment of \$10/monthly invoice, and an inventory control cost of \$40/yr.

ments in order to properly size a system.

#### Test laboratory example

Minnesota Valley Testing Laboratories (MVTL, New Ulm, MN) represents an example of the benefits of on-site gas generation. The laboratory provides environmental, agricultural, and energy testing services to industry. The laboratory was purchasing ultrahigh-purity hydrogen cylinders to supply fuel gas for FIDs and nitrogen phosphorous detectors (NPDs), make-up gas for electrolytic conductivity detectors (ELCDs), and carrier gas for GC columns. Safety was a concern and monitoring of the cylinders was an inconvenience. Technicians had to continually watch the cylinders. Hydrogen was used as a carrier gas for GC columns, and if it were to run out, the column would be damaged." Also, MVTL would occasionally get a contaminated cylinder, resulting in skewed analysis and delayed work.

The laboratory has purchased on-site generators to replace zero air cylinders for fuel air to FIDs, flame photometric detectors (FPDs), and NPDs; to replace ultrahigh-purity nitrogen cylinders for make-up gas for electron capture detectors (ECDs), NPDs, ELCDs, and FIDs; and to replace hydrogen cylinders for fuel gas

applications. An MVTL chemist indicated that the unit will have paid for itself in less than two years, after which the laboratory will be producing its own hydrogen at a fraction of the cylinder cost. Other benefits cited by MVTL include no longer having to handle and monitor cylinders. Low maintenance is also an advantage. Installation was easy: the generator was plugged in, water was added, and the instruments connected. The hydrogen generator also takes up less space.

#### Conclusion

Change is never easy and it is seldom comfortable, but change allows us to learn and improve, increase efficiencies and convenience, achieve better performance, reduce costs, and solve difficult problems. In this day of corporate reengineering and global competition, laboratories are experiencing a paradigm shift related to the use of compressed gases. This shift from high-pressure gas cylinders to on-site gas generators will result in reduced operating and overhead costs, positioning laboratories to be more competitive and successful.

In considering the shift to on-site gas generation, compressed gas users should use convenience, reliability, safety, and cost as primary criteria. The convenience, reliability, and safety of gas generators compare favorably to

in the midst of a" analysis, overnight, or during a weekend. other inconveniences include delayed delivery, inflexible delivery schedules, price increases, and long-term contracts. The procurement procedure within the customer's company may also result in unwanted delays.

The use of high-pressure compressed gas cylinders must be accompanied by a concern for safety. The primary safety issues are transportation, handling, storage, use, asphyxiation, toxicity, and flammability. Special precautions must be taken while handling high-pressure cylinders.<sup>4,5</sup> A dangerous situation can be created if a cylinder is dropped and the valve is broken off, potentially causing the cylinder to become a projectile.<sup>6</sup> Other potential hazards that are inherent in the storage of compressed gases include asphyxiation, combustion, and explosion. Various regulations by the Department of Transportation (DOT) and the Occupational Safety and Health Administration (OSHA) address the hazardous aspects of high-pressure gas cylinders.<sup>8,9</sup> Potential hazards in the "use of high-pressure gas cylinders are serious enough to warrant the use of warnings indicating that improper "use can result in serious injury or death.

There are disadvantages to the "use of compressed gas cylinders based on gas purity considerations. The gas purity will vary from one cylinder to another and it will also vary as the gas within the cylinder becomes used." Cylinders may be dirty, rusted, and contaminated, resulting in outgassing of contaminants from the cylinder walls. Compressed gas supplied in high-pressure cylinders can be tested and certified upon request, for an additional charge. If a cylinder is not certified, the customer has no confirmation that the actual purity meets the stated specification." The result of using a gas that has a purity below specification can be the costs associated with inaccurate analysis.

Many indirect or hidden costs in the use of high-pressure gas cylinders include the time and effort to change cylinders; generation and expedition of a purchase order: receipt of full cylinders and shipment of empty cylinders: payment of monthly invoices; and maintenance of a gas inventory for re-

Table 1

Cost component	Typical cost
Cylinder gas price	\$100
Cylinder demurrage (one month)	\$5.25
Labor cost to change cylinder	58.33
Order processing cost	510
Shipping cost	510
Invoice payment cost	\$10
Inventory control cost (monthly)	53.33
Total	5146.91

\*Gas cylinder price is based on typical ultrazero-grade air.

All indirect costs are typical for gas cylinder suppliers or calculated through activity-based costing of a manufacturing company.

ordering purposes. Consider the departments involved in this process: R&D, purchasing, shipping, accounts payable, and central supplies. These hidden overhead costs are generally accepted as part of the cost of operating a laboratory and should be taken into consideration during the process: of choosing a supply of compressed gases. Laboratory managers might be surprised if the full cost of compressed gas delivery was plainly visible.

All of the costs listed (direct and indirect) can be obtained either from the compressed gas supplier or through an activity-based costing approach to the purchasing company's overhead costs. This type of cost analysis is shown in *Table 1* for the purchase of a single cylinder of ultrazero-grade air used for one month, before replacement. This analysis shows that the hidden costs amount to an additional 47% of the actual purchase price of the gas delivered in the high-pressure cylinders.

#### *On-site generation of compressed gases*

In the past five years, the availability and "use of on-site compressed gas generators has increased significantly." This increase can be attributed primarily to the improvement of technologies used in the generation and purification of gases, the recent commercialization of many on-site generators, and the ever-increasing prices for compressed gases delivered in high-pressure cylinders.

The technologies contributing to the increased availability of point-of-use gas generators include membranes,

specialized adsorbents and catalysts, improved air compressor designs, and enhanced electronic controls. These technologies will continue to improve the availability, performance, and value of laboratory gas generators. The most influential of these technological developments is the "use of membranes and specialized adsorbents for the production of nitrogen gas. Joint ventures between industrial gas companies and chemical companies have resulted in successful R&D programs to improve the technology for on-site generation of nitrogen. On-site generation of nitrogen is available for purities ranging from 95% to 99.9995%. Other gases that can be reliably produced on site include hydrogen and various purities of air.

The effectiveness of on-site gas generation should be analyzed based on convenience, reliability, safety, and cost. The attractiveness of on-site gas generation depends greatly on the specific gas being used, the required purity, required flow rate, hours of operation per day, and local gas prices. For instance, on-site generation may not be an attractive alternative for an application requiring sporadic usage of research-grade nitrogen based on cost alone, whereas it will be attractive for purging an FTIR spectrometer or providing hydrogen fuel to a flame ionization detector (FID) that operates 24 hr day (based solely on cost).

Gas generators provide convenience through the elimination of reliance on an external delivery service. Once the on-site gas generator has been purchased and installed, delivery of the desired gas is automatic, reliable, and relatively inexpensive. Typical preventative maintenance is performed on an annual basis and requires little or no downtime.

Reliability of on-site generators is based on the operation of simple electromechanical components involved in gas purification. These components, such as pressure vessels, valves, timers, and heaters, have a history of reliability in industrial applications. The logistics of a delivery system have been eliminated so that reliability is based only upon the performance of the gas generator.

The purity of gas delivered by qual-

**high-pressure gas** cylinders. A simple, cost analysis utilizing activity-based costing techniques will show paybacks of less than one year for the change to on-site gas generation for many typical applications. Over the next decade, as this paradigm shift matures, laboratory managers and scientists will view the last few cylinders in their laboratory similar to the way that we all view the glass milk bottle.

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260 Neck Rd., Box 8223  
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Whatman Canada Ltd  
2495 Haines Rd.  
Mississauga  
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# The effect of detector gas purity on FID baseline stability

It is well accepted in the field of gas chromatography that the purity of gases utilized for the operation of the gas chromatograph will affect the accuracy of analyses, consistency of results, detector sensitivity, and column life as well as column performance. As a result, it is important that sources of gas used by the chromatographer meet the highest possible purity standards affordable in order to meet the demand to achieve higher sensitivities required for environmental analysis, quality control procedures, government regulations, as so forth. Traditionally, this source has been a high-pressure gas cylinder specified to a wide range of purities (99.99% to 99.9999%). More recently, gas generators have become available as a source of high-purity hydrogen (for carrier and fuel gas) and high-purity nitrogen (for make-up gas).

This paper describes the results of a performance study using the Whatman Model 75-34 Hydrogen Gas Generator (Fig. 1) and the Whatman Model 75-92 Ultra High Purity Nitrogen Generator (Fig. 2) (Whatman Inc., Haverhill, MA) to provide make-up gas and detector fuel for a GC flame ionization detector. The objective of the study was to demonstrate any effect of using hydrogen and nitrogen from the generators on the performance of the gas chromatograph. The results of the instrument operation using the generators as a source were compared to the results using the highest purity grade of cylinder gas (research grade) available. Baseline stability was the primary basis for comparison of the gases in the study.

Dr. Barry is a *Professor of Chemistry and Graduate Coordinator at the University of Massachusetts at Lowell, Dept of Chemistry, One University Ave., Lowell, MA 07854, U.S.A. tel.: 5-08-934-3669*

The chromatographer's concerns with the quality of the gases used in the analysis focus on impurities. Impurities in a chromatographer's gas supply can result in excessive noise, baseline drift, ghost peaks, column bleed, and reduced column life.<sup>1-3</sup> The specified purity of the gases supplied by the generators is 99.9995% for the nitrogen and 99.99999% for the hydrogen. These stated purities are sufficiently high that the gases should yield a baseline stability comparable to that achieved with the highest purity cylinder gases available, namely research grade.



Figure 1 Model 75-34 hydrogen gas generator

Table 1

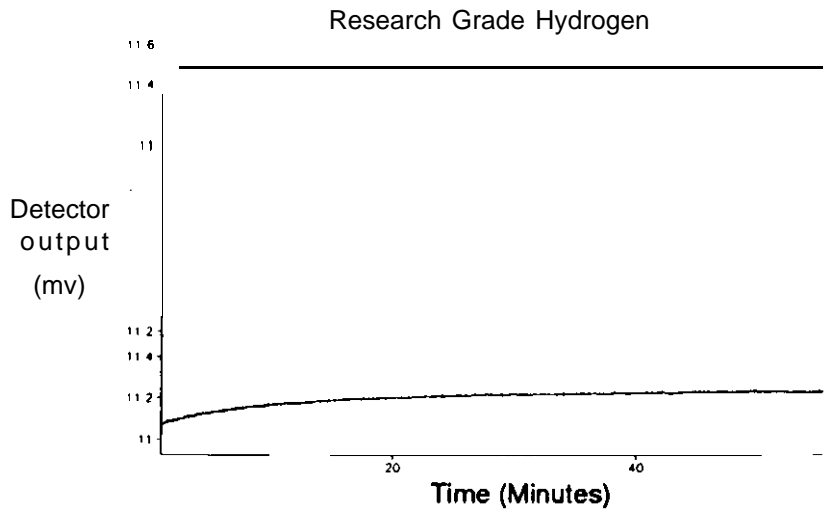
Gases used to establish baselines shown in Figure 3				
Gas function	Gas source	Specified purity	Flow rate (cm <sup>3</sup> /min)	Pressure (psig)
Fuel air	Whatman Zero Air Generator	<0.1 ppm total hydro-Carbons	300	35
Fuel Gas H <sub>2</sub>	Whatman 75-34 H <sub>2</sub> generator	99.99999%	40	22
Carrier gas (He)	Research-grade helium cylinder	99.9999%	0.7 23 cm/sec linear velocity	40
Make-up gas (N <sub>2</sub> )	Research-grade N <sub>2</sub> cylinder	99.9995%	30	20

Table 2

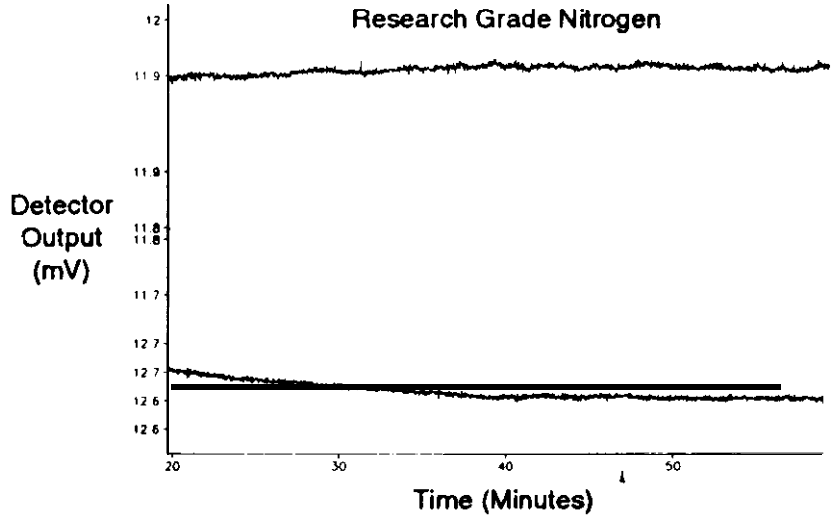
Gases used to establish baselines shown in Figure 4				
Gas function	Gas source	Specified purity	Flow rate (cm <sup>3</sup> /min)	Pressure (Psig)
Fuel air	Whatman Zero Air Generator	0.1 ppm total hydro-carbons	300	35
Fuel Gas (H <sub>2</sub> )	Research-grade H <sub>2</sub> cylinder	99.9999%	40	22
Carrier gas (He)	Research-grade helium cylinder	99.9999%	0.7 23 cm/sec linear velocity	40
Make-up gas (N <sub>2</sub> )	Whatman 75-92 N <sub>2</sub> generator	99.9995%	30	28
	research-grade N <sub>2</sub> cylinder			



**Figure 2** Model 75-92 UHP Nitrogen Gas Generator.



**Figure 3** Gas chromatograph baselines using a 75-34 hydrogen generator and a research-grade cylinder as fuel sources.



**Figure 4** Gas chromatograph baselines using a 75-92 UHP nitrogen generator and a research-grade cylinder as make-up gas sources.

### Experimental

This gas chromatography performance study was conducted at the University of Massachusetts, Lowell, using a 5890A GC (Hewlett-Packard Co., Palo Alto, CA) with flame ionization detector. The column used during the study was a HP-1 fused silica capillary column, 30 m in length, 0.07-mm i.d., with a film thickness of 0.25  $\mu\text{m}$ , and the column

was operated under isothermal conditions at 100°C at a linear velocity of 23 cm/sec. The split ratio used in the performance testing was 100/1. The instrument used two sets of conditions in order to obtain a set of baselines to show the effect of the hydrogen generator as a source of fuel gas (Figure 3) and a set of baselines to show the effect of the

UHP nitrogen generator as a source of make-up gas (Figure 4). The operation of the instrument to obtain the baselines shown in Figure 3 used the gases as described in Table 1. The operation of the instrument to obtain the baselines shown in Figure 2 used the gases as described in Table 2.

## Results

Figure 3 shows the effect of using fuel hydrogen supplied by a 75-33 hydrogen generator on baseline stability. The baseline generated is compared to that generated while using a research-grade cylinder of hydrogen as the fuel gas source. Figure 3 shows that there is not a distinguishable difference between the two baselines.

Figure 4 shows the effect of using make-up nitrogen gas supplied by a 75-92 UHP nitrogen generator on baseline stability. The comparison of the baseline generated while using make-up gas from a research-grade cylinder shows that the two gas sources are indistinguishable.

## conclusion

A GC was used to compare the baseline stability of a flame ionization detector while using a gas generator as the source of **detector** fuel and make-up gas with the baseline stability obtained while using corresponding research-grade cylinder gases. The results demonstrate that the 75-34 hydrogen generator (used as a source of detector fuel) and the 75-92 UHP nitrogen generator (used as a source of make-up gas) will provide the chromatographer with GC baseline stability equivalent to that achieved with the highest purity grade of gas cylinder available, while offering the inherent advantages of eliminating high pressure gas cylinders (such as safety, convenience, and cost).

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## For Additional Information

Whatman Inc.. also manufactures and markets a complete line of Whatman Gas Generators including: Ultra High Purity Nitrogen Generators, Gas Generators for FT-IR Spectrometers and NMR's, TOC Gas Generators, and Complete Systems with Oil-less Compressors. All generators are designed to enhance instrument accuracy and performance, and increase laboratory efficiencies by automating gas delivery systems. Detailed information is available by contacting Whatman Inc., 260 Neck Road, Haverhill, MA 01635. Call toll-free at 1-600-343-4046, or fax 1-506-374-7070.



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260 Neck Rd., Box 8223  
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800-343-4048 or 978-374-7400  
Fax 978-374-7070

**Whatman Canada Ltd**  
2495 Haines Rd.  
Mississauga  
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# Whatman Zero Air Generators

Models available to service up to 100 FIDs



High purity zero air generator ensures a consistently flat baseline

Produces gas equivalent to UHP Zero Air cylinder gas at a fraction of the cost - detailed purity graph and lights included

Eliminates the need to recalibrate instruments after replacing empty cylinders of gas

Compact design frees up valuable laboratory floor space - system is wall or bench mountable.

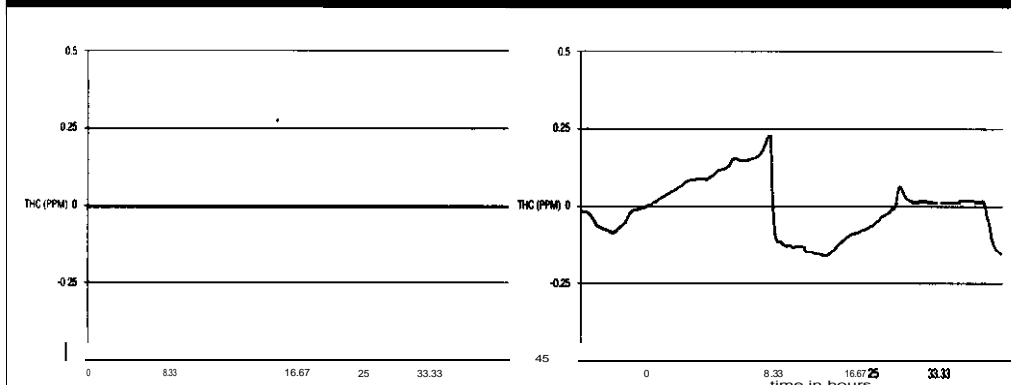
Operates at low pressures, from 40 to 125 psig - much safer than high pressure gas cylinders

Model Number	Number Of FIDs'
75-83	up to 3
76-803	up to 11
76-807	up to 23
76-818	up to 60
76-830	up to 100

based on 300 ccm fuel air rate

Restek (U.S.): 110 Benner Circle  
Bellefonte, PA 18823  
Australian Distributors - Chromalytic Technology  
Fax : +61 3 9761 1169

## Baseline Comparison



The Chromatograms (right) compares baselines produced by a Whatman Zero Air Generator (top) and bottled fuel air (bottom). The baseline produced by the Whatman Generator is very flat, with no fluctuations or peaks. In comparison with the chromatogram of the bottled air fuel supply, which has many peaks ranging from .25 ppm to -.25 ppm.

## Principal Specifications

Whatman Models 75-83, 76-803, 76-807, 76-818, 76-830			# of FIDs
Max Zero Air Flow Rate	75-83 76-803 76-807 76-818 76-830	1.0 lpm 3.5 lpm 7 lpm 18 lpm 30 lpm	up to 3 11 23 60 100
Outlet Hydrocarbon Concentration (as methane)		< 0.1 ppm	
Min/Max Inlet Air Pressure		40 psid/125 psiQ	
Max Inlet Hydrocarbon Concentration (as methane)		100 wpm	
Pressure Drop at Max Flow Rate		4 psid	
Max Inlet Air Temperature		78°F (25°C)	
Inlet/Outlet Ports		1/4" NPI (femals,	
Start-up Time for Specified Hydrocarbon Concentration (as methane)		45 minutes	
Electrical Requirements	75-83 76-803 76-807 76-818 76-830	120 VAC/60Hz 0.5 amps 120 VAC/60 Hz, 3.5 amps 120 VAC/60 Hz 3.5 amps 120 VAC/60Hz 3.5 amps 120 VAC/60 Hz 3.5 amps	
Dimensions	75-83  Other Models	10" w x 3" d x 12" h (25cm x 8cm x 30cm, 11" wx 13" d x 16" h (27cm x 34 x 42cm)	
Shipping Weight	75-83 Other Models	7 lbs (3kg) 41 lbs (19 kg)	

## Ordering Information for assistance, call 800-356-1688 or 814-353-1300

Description	Model Number (Restak Cat.#)
Zero Air Generator	75-83 (20684) / 76-803 (20660) / 76-60 (20661) / 76-818 (20682) / 76630 (20683)
Maintenance Kit for Model 75-83	MK75.83 (21646)
Maintenance Kit for Models 76-803, 76-807, 76-818, 76-830	MK7840 (21647)



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# Whatman Ultra High Purity Nitrogen Generator

Models 76-92, 76-94, 76-96 (Flow Capacity: 1.1 lpm to 2 lpm)



- Pays for itself in 1-2 years
  - Install it once - uses compressed air and one 120 VAC/50 Hz outlet
- w Output purity exceeds UHP cylinder gas specifications at a fraction of the cost (Models 76-92 and 76-94)
- - Safe, reliable, low maintenance

A

All the generators are used for evaporating solvents, switching valves, purging chambers and many other applications where an inert gas supply is required. The generators are ideal for general laboratory nitrogen gas supply. The 76-92 and 76-94 UHP Nitrogen Generators provide a continuous supply of dry, ultra high purity nitrogen. The generators are ideal for providing carrier gas to gas chromatograph columns. They are also well-suited for use with packed column instruments. The 76-96 Nitrogen Generator provides a continuous supply of dry, 99.99% pure nitrogen for laboratory use.

## Principal Specifications

Model	76-92/76-94	76-96
Max Nitrogen flow rate	See Flow Table	2 lpm
Nitrogen Purity	99.9995%	99.99%
Max Nitrogen output pressure	See Table	90 psig
CO concentration	< 1.0 ppm	NA
CO <sub>2</sub> concentration	< 1 ppm	< 1 ppm
H <sub>2</sub> O concentration	< 1 ppm	< 100 ppm
H <sub>2</sub> O Concentration	-2 ppm	-2 ppm
Hydrocarbon concentration (1)	< 0.1 ppm	NA
Argon concentration (2)	0.9%	0.9%
Min/Max inlet pressure	60 psig / 125 psig	75 psig / 120 psig
Recommended inlet temperature	76°F (25°C)	76°F (25°C)
Ambient operating temperature	60°F-100°F (16°C-36°C)	40°F-100°F (4°C-38°C)
Max air consumption	42 lpm (1.5 scfm)	43 lpm (1.5 scfm)
Inlet connection	1/4" NPT (female)	1/4" NPT (female)
Outlet connection	1/8" compression	1/8" NPT (female)
Electrical requirements (3) (4)	120 VAC 60Hz	120 VAC 60Hz
Dimensions	12"wx16"dx3"h (30cm x 41cm x 69cm)	12"wx16"dx35"h (30cm x 41cm x 69cm)
Shipping Weight	115 lbs. (52 kg)	115 lbs. (52 kg)

- 1 Model 76-92 does not remove hydrocarbons
- 2 Purity specification for Nitrogen does not include Argon concentration
- 3 Power Consumption is as follows: Model 76-92= 25 Watts, Model 76-94= 700 Watts, Model 76-96= 25 Watts
- 4 Global power configurations available

## Flow Table

Inlet Air Pressure (psig)	Max Outlet Flow (cc/min.)	Max Outlet Pressure (psig)
Model 76-92 and 76-94		
125	1100	65
110	1000	75
100	900	65
90	600	60
60	700	50
70	600	45
60	500	35
Model 76-96		
75-120	2000	90

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Tb-

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**RESTEK**

## Ordering Information for assistance, call 800-356-1688 or 314-355-1300

Description	Model Numbers	Restek
High Purity Nitrogen Generator	76-96	21654
Ultra High Purity Nitrogen Generator	76-92 and 76-94	21653 and 20697
Indicating Oxygen Trap (box of 2)	72092	
Auxiliary Prefilter	A912A-DX	
Maintenance Kit	MK7692	21649

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# Whatman Nitrogen Generation Systems

Models 75-72, 75-720, 75-880



- Nitrogen purity up to 99.5%
- Up to 150 SCFH per minute of continuous output capacity
- Safe, operate at low, regulated pressures
- Produce a constant gas supply to maintain consistent analytical results
- Payback period of 1-2 years
- Very reliable, no moving parts
  - No more time consuming cylinder Changes-install just once for worry-free operation
- Ideal for API (MS) as a curtain or shield gas

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814-353-1300 \* NNN-35N-1NNN

**Restek France:**

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Resl8k OmkH:

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49-8198-85130

**TkamsR&ekUK, LM :**

Fairacres Industrial Cents - Dedwth Rd.  
Windsor Berkshire SL44LE  
01753 624111

## Nitrogen Purity/Flow Chart

Flow measured in scfh at Indicated Operating Pressure, psig at 65°F or 20°C)

N2 Purity - Models 75-72, 75-720

%	145	125	110	100	90	80	70	60
99.5	18	15	13	12	110	7	5	4
99	31	26	22	20	17	14	11	9
98	43	36	31	28	24	20	16	13
97	51	43	37	34	29	24	20	16
96	63	54	47	42	38	30	24	19
95	78	67	58	52	45	38	28	25
N2, Purity- Models 75-880								
99.5	150	130	110	100	80	60	(consult factory)	

## Principal Specifications

Model	75-72/75-720	75-880
Nitrogen Purity	95 - 99.5%	99.5%
Atmospheric Dewpoint	-58°F(-50°C)	-58°F(-50°C)
Suspended Liquids	None	None
Particles >0.01µm	None	None
Commercially Sterile	Yes	Yes
Min./Max. Operating Pressure	60/145 psig	60/145 psig
Max. Press. Drop @ 95% N2 Purity, 125 psig	10 psig	15 psig
Max. Recommended Inlet Temperature	<77°F(25°C)	<77°F(25°C)
Max. Ambient Operating Temperature	110°F(40°C)	110°F(43°C)
Air Consumption @ Max. Flow, 95% Purity	21.8 scfm(102 lpm)	consult factory
Electrical Requirements	75-72 none	120 VAC/60 Hz
Inlet/Outlet Ports	1/4" NPT	1/2" NPT
Shipping Weight	72-75:75lbs.(34kg), 75-720:80lbs.(35kg)	250 lbs. (114 kg)
Oxygen Analyzer	Included with Models 75-720 and 75-880	
Dimensions	50"h x 16"w x 16" d (127cm x 40cm x 40cm)	69"h x 24"w x 20"d (175cm x 61cm x 51cm)

## Ordering Information for assistance, call 800-356-1688 or 814-353-1300

Model 75-72	Restek Cat.# 20677
Model 75-720	Restek cat., 21652
Model 75-880	Pease Call
<b>Replacement Parts</b>	<b>75-72/75-720</b> 75-880
1st Stage Prefilter Cartridges	100-12-DX 100-18-DX
2nd Stage Prefilter Cartridges	100-12-BX 100-18-BX
3rd Stage Prefilter Cartridges	NA 100-25-BX
Final Membrane Filter Cartridges	GS-100-12-95 GS-100-25-95
Galvanic Cell	72695 (75-720 only) 72595
Activated Carbon Filter	NA
Filter Maintenance Kit	Restek cat.+ 21648 MK7572."" 75478""

\*\*Maintenance Kit includes 3 each of the 100-12-DX, 100-12-BX, and GS-100-12-95

\*\*\*Maintenance Kit includes 3 each of the 100-18-DX, 100-18-BX, 100-25-BX, and GS-100-12-95

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