Supelco™ Gas Purifiers for Total Protection of GC Systems

Contaminants entering a carrier gas stream can adversely affect the performance of a GC system. To prevent damage and interference caused by water, oxygen, and other critical contaminants, purification of gases entering a GC system is recommended.

Key Words:
- gas purifier
- contaminants
- gas chromatography

Contaminants such as water and oxygen can enter carrier or other gas streams, and adversely affect the performance of a gas chromatographic system. Carrier gas — one of the more critical gases entering the GC system — normally contains less than 10ppm of O_2 and H_2O. Even though these concentration levels are normal, they can be detrimental to a GC system. And a *contaminated* cylinder of carrier gas will contain even higher concentrations.

To prevent damage and interference caused by O_2, H_2O, and other critical contaminants, most manufacturers of gas chromatographs and ultrasensitive detectors recommend purification of gases entering a GC system. Supelco's full line of purifiers meet your gas purification needs:

- Molecular Sieve Drying Tube — easily handles *excessive* water contamination
- High Capacity Gas Purifier — irreversibly removes O_2, H_2O, CO, and CO_2
- OMI™ Indicating Purifier — removes all chromatographically critical impurities at ambient temperature and indicates its level of exhaustion
- Supelcarb™ HC Trap — removes hydrocarbons

Regardless of how you use our purifiers – individually or as components in a total purification system – they will completely and reliably remove oxygen, water vapor, and other critical contaminants from carrier and other inlet gases.

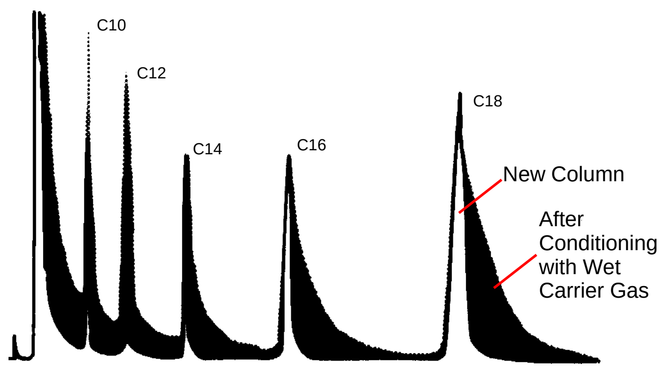
How Different Columns React to Contaminants

Packed Columns

At elevated temperatures, packed columns' stationary phases — such as polyesters (DEGS, EGS, etc.), polyglycols (CARBOWAX®, UCON®, FFAP, SP™-1000), and polyamides — are susceptible to both oxidation and hydrolysis (Figure A). Silicone phases will also react with oxygen when the temperature is above 250°C.

Columns employing these stationary phases perform satisfactorily for months with pure carrier gas. But if impure gas is used, even at typical GC operating temperatures, the column may quickly deteriorate. This deterioration (which may be indicated when the packing at the front end of the column turns brown) typically causes loss of column efficiency, shifting retention times, and peak tailing.

Figure A. Wet Carrier Gas Adversely Affects Packed Columns



10% SP™-1000 on 100/120 Chromosorb® W AW

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Capillary Columns

Capillary columns do not present a direct indication of deterioration (such as color change). But a capillary column's stationary phase is quite vulnerable to water and oxygen contamination. The constant presence of oxygen and water in your gas stream can damage your column's phase and alter the chromatography. Progressive oxygen and water damage to the stationary phase shortens column life (Figure B).

Capillary columns containing bonded, crosslinked phases are not a perfect solution to the problem of impure gas. These columns may handle short-term exposures to oxygen and water contamination, but extended exposure will still damage the phase and affect your chromatography.

Figure B. Oxygen Degrades Thin Phases of Capillary Columns

Column: **Carbowax® PEG 20M, 30m x 0.25mm ID, 0.25µm film**
Oven: 155°C
Carrier: helium, 20cm/sec, set at 155°C
Det.: FID, splitter vent and septum purge turned off for 25 min

Column Exposed to 100µL Air

Phase oxidation was monitored by observing the baseline as air was injected into the column. The full scale baseline deflection, followed by baseline instability, reveals that the phase is severely degraded.

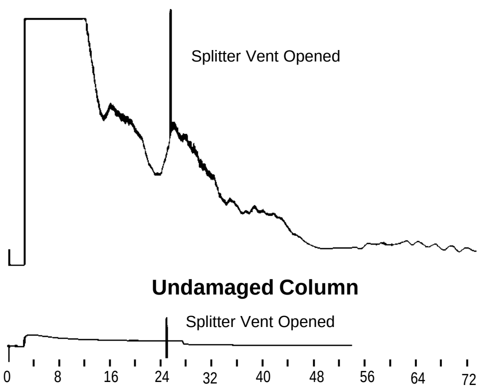
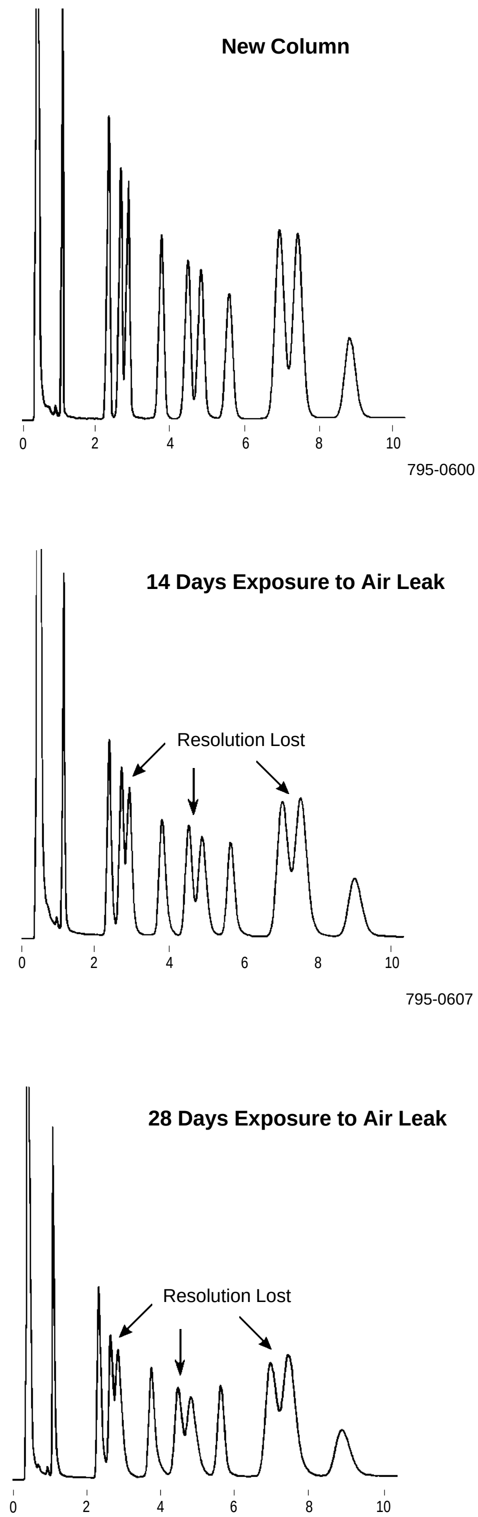


Figure C. Rapid Degradation of Adsorption Chromatography Columns

Packing: **Carbopack™ graphitized carbon modified with polyethylene glycol**
Column: **2m x 2mm ID glass**
Oven: 225°C
Carrier: nitrogen, 125mL/min
Det.: FID
Inj.: phenols mixture



Adsorption Columns

Adsorption chromatography columns — such as molecular sieve, silica gel, and alumina — deteriorate as they adsorb water from the carrier gas. Retention times will decrease as the moisture content of the adsorbent increases. Carbon supports, such as Carbosieves and Carbopacks, are attacked by O_2 at temperatures above 200°C (Figure C). And porous polymer packings, such as Chromosorb® 102 and Porapak® Q, are quite susceptible to oxidation.

The Problem in Gas Purification

Some carrier gas purifying devices — due to flaws in their design — may actually *add* oxygen and water to your carrier gas (Figure D). We tested devices made from metal, glass, or plastic tubing, with Swagelok®, Teflon®, or O-ring end seals.

We tested these different types of construction by passing highly purified gas (less than 1ppm each of oxygen and water) through each purifier. If gas leaving the devices contained oxygen or water levels greater than 1ppm, contaminants were obviously entering the system.

Not only did most of the moisture-removing devices allow moisture into the carrier gas, many of them also introduced oxygen. Water and oxygen levels were highest, by far, in the effluent gas from plastic tubes with O-ring seals (Figure E). These devices introduced an average of 16ppm water and 5ppm oxygen into the previously pure carrier gas. In some cases, contamination levels approached 100ppm water and 15ppm oxygen. Ironically, devices that seal with O-rings are among the most commonly used.

Figure D. Leaks and Permeation Contribute to Carrier Gas Contamination

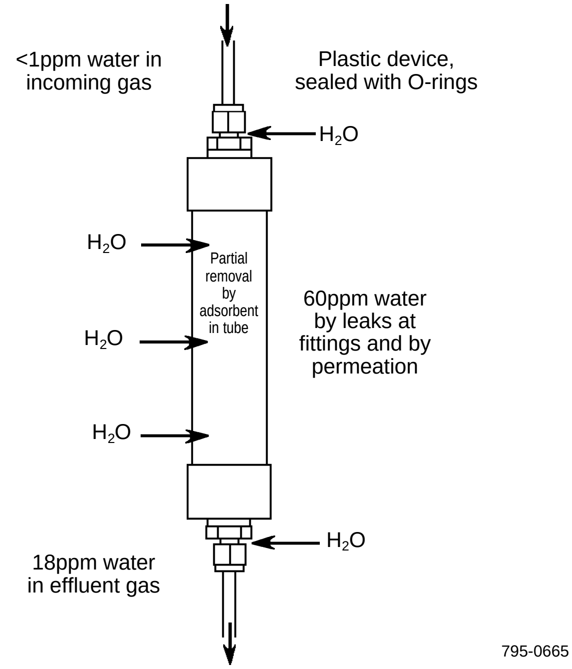
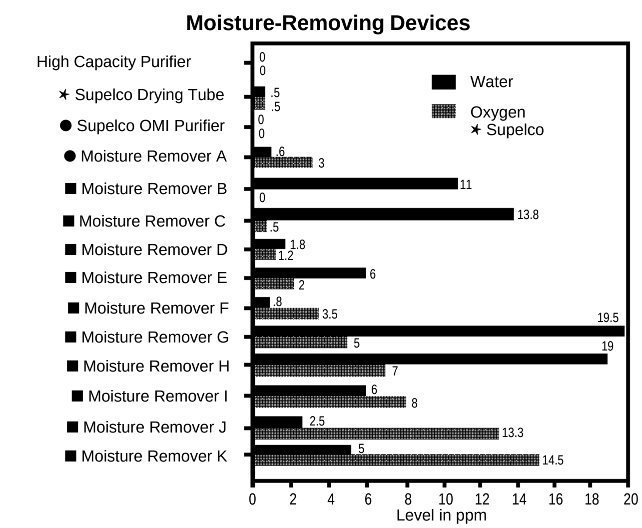
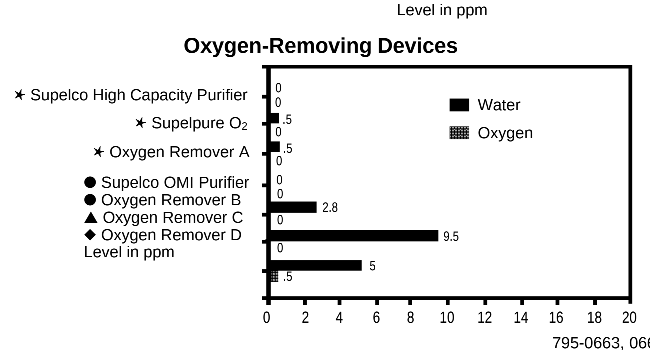


Figure E. Purity of Carrier Gas Passed Through Devices Designed to Remove Contaminants

	Tubing	Seals
✶	Metal	Swagelok
●	Glass	Teflon
■	Plastic	O-Rings
▲	Metal	O-Rings
◆	Plastic	Glass/O-Rings





The Solution to Gas Purification

To fully protect your GC columns and detectors from oxygen and water damage, you should use a gas purification system specifically designed to prevent leaks *and* permeation — the Supelco Gas Purification System.

This system consists of three highly effective purifiers:

- A Supelcarb Hydrocarbon Trap
- A Supelco Molecular Sieve Drying Tube
- A Supelco High Capacity Heated Gas Purifier

Plus:

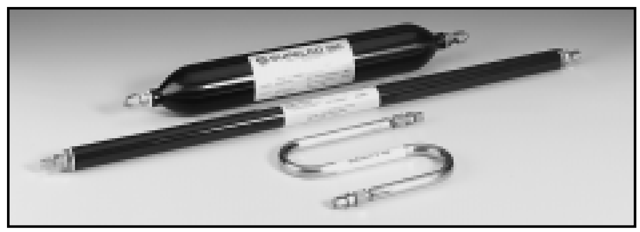
- An OMI Indicating Gas Purifier

For details on how to assemble this system, request Bulletin 918.

Supelco Molecular Sieve Drying Tube

If your analysis requires protection from water vapor contamination, connect the *Supelco Molecular Sieve Drying Tube* (Figure F) to your gas line. The drying tube will remove water and heavy hydrocarbons from compressed air used with flame detectors.

Figure F. Molecular Sieve Drying Tube



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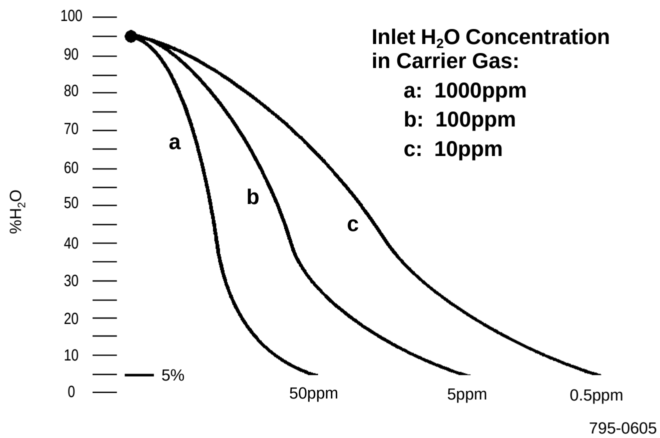
The drying tube, which contains molecular sieve 5A, can also be used on electrolytic hydrogen, in-house nitrogen, or other gas lines where moisture content could be high. In these circumstances, molecular sieve provides a preferred first line of protection. And because Supelco Molecular Sieve 5A offers greater *working* capacity, it is a better choice than molecular sieve 4A.

The greater working capacities of molecular sieve 5A are not the only advantages of this drying tube. Its two-foot length – twice that of some other molecular sieve tubes – is important because of the mechanism by which water adsorbs to molecular sieve.

Passing a fluid or gas that contains an adsorbable compound through a molecular sieve bed establishes a concentration gradient. This gradient is most conveniently represented by a sigmoidal curve known as a mass transfer front. The length of bed that contains a major portion of this mass transfer front is called the transfer zone. Conventionally, a transfer zone begins where the molecular sieve reduces the inlet concentration of the adsorbate by 5%, and ends where the inlet concentration is reduced by 95%.

The length of the mass transfer zone primarily depends on the velocity of the carrier gas stream, and on the concentration of water in the carrier gas and on the concentration of water in the carrier gas entering the molecular sieve bed (Figure G). It also depends on the mass transfer of the adsorbable compound (adsorbate) from the carrier gas stream to the molecular sieve bed.

Figure G. Molecular Sieve Mass Transfer Zone (Theoretical Mass Transfer Zone)



Although reduction of water impurities to concentrations <10ppm is extreme for most specialty gases, this degree of removal is often required under analytical conditions. Removal of water impurities to concentrations under 10ppm requires a mass transfer zone that is several times longer than described by conventional definition. The added length is required because, as adsorbate is transferred from carrier phase to molecular sieve, the rate of transfer decreases (sometimes dramatically) as adsorbate in the carrier gas phase-molecular sieve matrix approaches an equilibrium concentration. Longer mass transfer zones in the longer tube better accommodate a decreased rate of transfer. The greater accommodation increases the tube's efficiency, improving its performance under extreme removal conditions.

The mass transfer zone is progressively displaced from inlet to outlet of the molecular sieve bed. Displacement gradually occurs as that portion of the molecular sieve bed containing the mass transfer zone becomes equilibrium saturated with the adsorbate.

When the leading edge of the mass transfer zone reaches the bed's outlet, the bed is considered to be exhausted.

The additional length of Supelco's molecular sieve drying tube also provides greater sequential space to handle more mass transfer zones. The increased number of theoretical mass transfer zones means a greater working capacity for the drying tube.

Two other versions of the molecular sieve drying tube also are appropriate for GC use. The "S"-shaped trap in Figure F combines the long (24 inches) bed length needed for effective removal of water vapor with a convenient shape. The large trap contains 750cc of adsorbent, compared to 200cc of adsorbent in the original model, making it suitable for use in gas lines supplying multiple chromatographs, or for analysts who want to minimize the number of times they change purifiers.

Other advantages of the molecular sieve drying tube include the tube's inert, metal construction, that prevents oxygen and water diffusion or leakage into the carrier gas. And because of the greater working capacity and efficiency of Supelco Molecular Sieve 5A, it can reduce water impurities in carrier gas to final concentrations less than 0.1ppm.

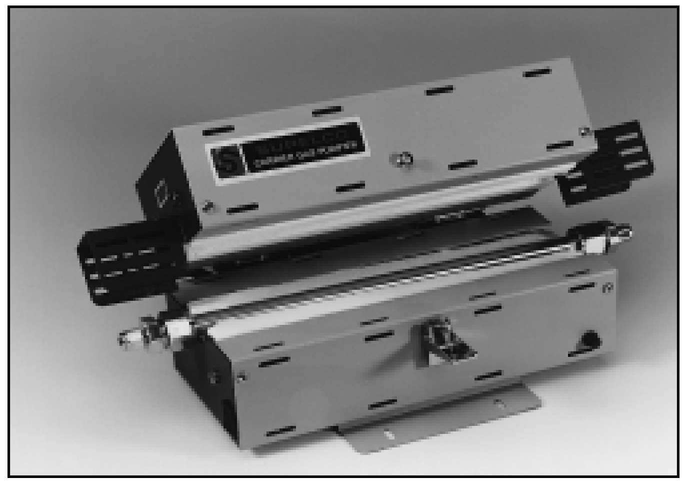
We also offer an economy-priced trap, suitable for reducing water contamination in air and actuator gases (see **Ordering Information** on page 8).

Supelco High Capacity Heated Gas Purifier

The Supelco High Capacity Heated Gas Purifier (Figure H) consists of a compact, all-metal, 10" long furnace that holds a stainless steel converter tube. The converter tube comes with Swagelok® fittings and contains a *gettering* material that, when heated, chemically reacts with oxygen, water, carbon monoxide, carbon dioxide, and hydrogen. Most other purifiers remove only oxygen *or* water from carrier gas and *do not* contain material that chemically and irreversibly reacts with these contaminants.

The chemical reaction between the gettering material and O_2 and H_2O ensures that these contaminants cannot return to the gas stream – not even with oven temperature fluctuations or when the material's capacity is exhausted. The reaction occurs uniformly along the converter tube until the material is completely expended. No unreacted material is wasted. When the gettering material is exhausted, the converter tube is easily replaced.

Figure H. High Capacity Heated Gas Purifier



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The Supelco converter tube has an all metal construction that eliminates diffusion of oxygen and water into the carrier gas. Many purifiers contain tubes constructed of various types of plastic. But plastic tubes have problems with diffusion of ambient oxygen and water vapor. They can also offgas hydrocarbons into the gas stream. Thus, other purifiers may actually introduce contaminants into the carrier gas.

The capacity of the Supelco converter tube is 14 liters of O_2 or 34 liters of H_2O vapor. When a cylinder of carrier gas contains either oxygen or water – or some combination – at a concentration of 50ppm, the gas is 99.995% pure. The converter tube will reduce oxygen and water impurities in up to 25 cylinders of this carrier gas to concentrations of less than 0.1ppm.

The high capacity purifier also handles flow rates up to 1100mL/minute. And with the appropriate gas line configuration, a single high capacity purifier can bulk purify gas for several gas chromatographs.

OMI Indicating Purifier

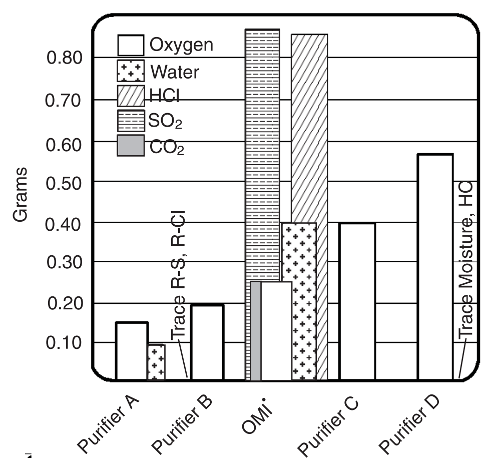
The OMI Indicating Purifier simultaneously and irreversibly reduces oxygen and water to less than 10ppb in a carrier gas stream. It is available in two sizes: the OMI-2 purifier removes up to 0.16 grams of oxygen or 0.2 grams of water, the OMI-4 removes up to 1.19 grams of oxygen or 1.4 grams of water. These capacities equal or exceed those of many non-indicating ambient temperature purifiers (Figure I).

The OMI Indicating Purifier minimizes potential oxygen and water breakthrough by handling oxygen surges of several hundred ppm, or carrier gas delivery rates up to 1000cc/minute (or 150psig). This purifier also removes carbon dioxide, carbon monoxide, most sulfur compounds, most halogen compounds, alcohols, phenols, and other trace impurities. Removal of these contaminants is particularly important when using ultrasensitive Hall detectors, electron capture detectors, or GC/MS systems.

The OMI purifier has an inert glass body that prevents diffusion of ambient oxygen and water into the carrier gas stream. The inert glass body also eliminates unacceptable background noise that can adversely affect highly sensitive detectors.

The main reason for this purifier's effectiveness is Nanochem® resin, a proprietary material licensed exclusively to Supelco for use in chromatographic applications. Contaminants, even at concentrations below 1ppm, progressively change the resin's color from black to light brown. Because Nanochem's color change can be seen at a glance through the OMI purifier's clear glass body and plastic safety guard, remaining purification capacity can be easily monitored. When the color change reaches the replacement mark, simply change the tube.

Figure I. Comparison of Ambient Temperature Purifiers



•Also purifies CO, R-S, R-X, C=O, ROH.
Capacities not additive.

Comparison based on OMI-1 tube (capacity: 0.25g oxygen or 0.4g water), now available only as a replacement tube (no fittings).

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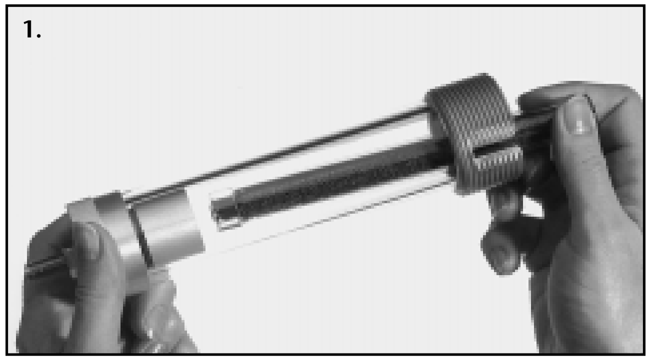
The OMI purifier maintains leak-free connections with specially designed seals. During shipment or storage, foil seals on each end of the purifier prevent oxygen or water from contaminating the resin. Beveled thorns inside the holder fittings pierce the foil seals when the OMI purifier tube is installed. This produces a closed system for the carrier gas flow, assuring that the purifier's resin is not exposed to ambient contaminants.

Replacement of spent tubes is easy (Figure J). Simply unscrew the end assembly from the tube holder and replace the old OMI tube. The design prevents air from entering the new tube during installation, preserving the resin.•

By itself, the OMI purifier will remove all critical carrier gas contaminants, providing a carrier gas purity that is better than most certified gases (Figure K). But connecting the OMI purifier downstream from a Supelco High Capacity Heated Gas Purifier produces a purification system specifically designed to economically give maximum gas purity. This total purification system provides a quality gas for superb FID and TC detector response. It also offers ppt detection capability with ECD, Hall, GC/MS and ultrasensitive detectors.

Figure J. Replacement of OMI Tube Is Easy

1.



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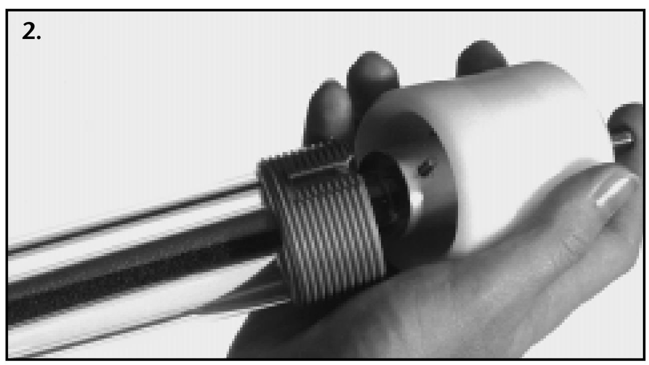
2.



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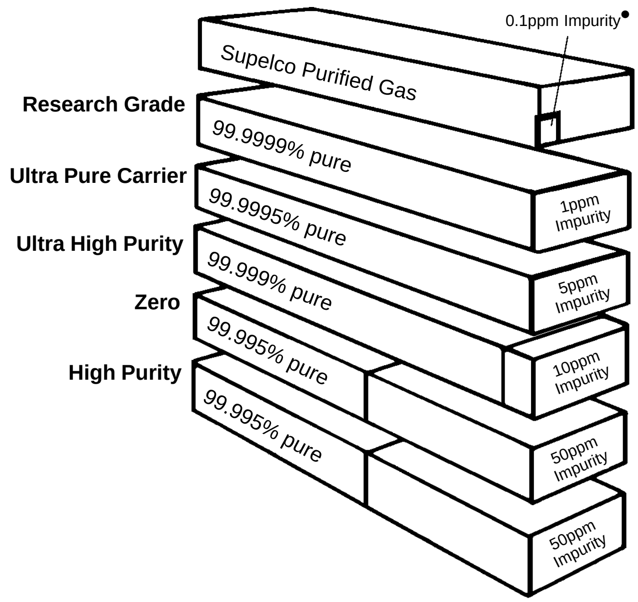
• Note: We do not recommend storing unused OMI tubes for more than six months.

Figure K. Supelco Purification System Reduces Chromatographically Critical Impurities to Lowest Concentrations



•Impurities include O_2, H_2O, CO_2, CO, and halogen and sulfur compounds.

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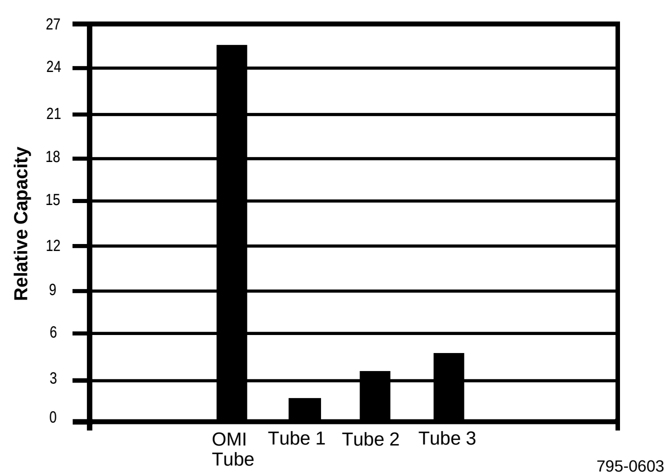
All Indicating Tubes Are Not Alike

Do not confuse the OMI Indicating Purifier with green oxygen indicating tubes. These green tubes, containing manganese oxide (MnO), efficiently indicate *only* oxygen. But they are seriously affected by oxygen concentration surges above 10ppm. These surges swamp the MnO and allow oxygen to break through to the GC system. And because these green tubes have very low capacities, they only signal an *already existing* problem. They do not warn of an impending problem you can ward off. Nor do they offer any protection against carrier gas impurities other than oxygen. The OMI purifier does not have capacity problems (Figure L) and warns you of impending difficulties *before* your column is damaged.

Figure L. An OMI Tube Traps More Oxygen Compared to Green Indicating Tubes



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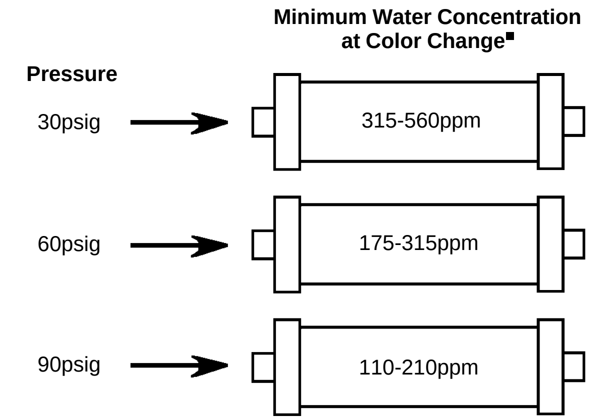
Be careful if your purifier or drying tube contains a blue indicator. The blue color comes from anhydrous cobalt chloride, which turns pink when exposed to water. But the moisture level required to hydrate the cobalt chloride significantly exceeds what a GC system will tolerate (Figure M). This leaves a range of moisture levels between what will be tolerated by the GC system and what will change the cobalt chloride's color. You could operate your system under potentially damaging conditions without realizing it. Absence of color change could simply mean there is insufficient water in the carrier gas to hydrate the cobalt chloride. This level of performance provides adequate protection only for actuator gas or air supplied to a detector. If you require a purifier to indicate exhaustion, the OMI purifier is your only choice.

Figure M. Cobaltous Chloride Moisture Indicators Do Not Protect Your Column



▪Ambient temperature

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Hydrocarbon Traps

Hydrocarbons can contaminate carrier gas, air, or fuel gas lines, creating unstable baselines and other problems. Address this contamination with a Supelcarb HC Hydrocarbon Trap. (Figure N). Supelcarb HC adsorbent has twice the trapping ability of the activated charcoal in other traps, ensuring the best performance of your system for the most demanding analyses. As Figure N shows, the Supelcarb HC Hydrocarbon Trap, like our Molecular Sieve Drying Tube, is available in several configurations and adsorbent volumes.

Relative to the Supelcarb HC Hydrocarbon Trap, the Supelpure™-HC Hydrocarbon Trap is lower in cost, but also is a less efficient trap. The activated charcoal it contains will perform effectively if the total hydrocarbon concentration in the incoming gas is below 10ppm.

Figure N. Supelcarb HC Trap

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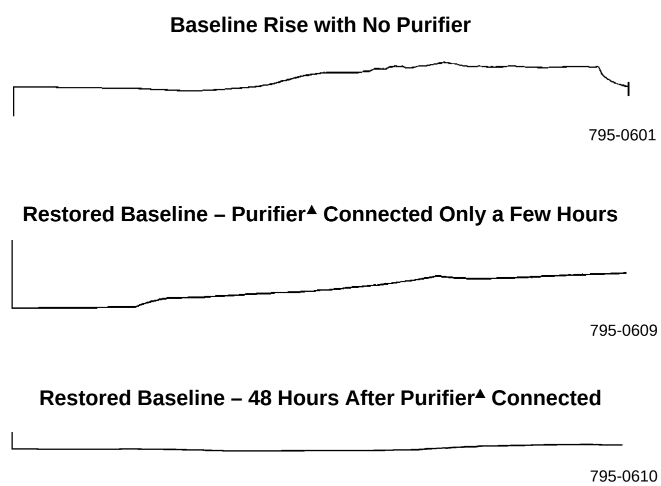
How Contaminants Affect Detector Performance

Columns are not the only component of a GC system that can be adversely affected by oxygen, water, and other chromatographically critical contaminants. These contaminants can also interfere with the performance of ultrasensitive detectors, causing chromatographic noise and baseline instability (Figure O). Important detector components may be permanently damaged.

Oxygen and water compete for the free electrons in electron capture and helium (argon) ionization detectors, causing lower detector responses for analyzed species. With Hall® detectors, oxygen and hydrogen contaminants appear as chromatographic noise and baseline instability. Trace H_2O and O_2 contamination in the carrier gas oxidizes the filaments of thermal conductivity detectors, resulting in a noisy baseline, low sensitivity, and shorter detector life. And although most mass spectrometer and analyses are performed at a mass range above that of O_2, water and oxygen can still reduce a mass spectrometer's sensitivity and shorten the life of the MS filament.

Figure O. Supelco Purifiers Reduce Detector Interference that Causes Baseline Rise

Oven: 150°C (4min) to 310°C at 16°C/min (5 min)
Det.: ECD, 350°C
Carrier: nitrogen, 60mL/min



▲OMI purifier on make-up gas line.

Packard Zero Air Generator

The Packard Zero Air Generator (Figure P) produces ultra-high-purity (UHP) grade, hydrocarbon-free air using a standard compressed air supply. Models 2500 and 2501 produce UHP air at continuous flow rates up to 2500cc/min, at pressures up to 125psig, with less than 0.1ppm total hydrocarbons. Models 1000 and 1001 are smaller, lighter and less expensive versions of Models 2500 and 2501. The maximum flow is 1000mL/min and the quality of the finished gas is equal to that of the Model 2500/2501.

Figure P. Packard Zero Air Generators

995-0116

Impurities in fuel gases used with flame ionization detectors (FID) affect the performance of the detector, particularly when operating at high sensitivity. The Packard Zero Air Generators are recommended for use with FID detectors because the improved signal to noise ratio and baseline stability allow lower detection limits, making more sensitive analyses possible.

The system consists of three stages: a 0.5µm coalescing inlet filter that removes particles, oil, and water, a heated catalyst that removes hydrocarbons, and a 0.01µm cellulose fiber outlet filter that removes any residual particulate material from the finished air stream. Minimal maintenance is required. Inlet and outlet filters should be cleaned twice per year and should be changed every two years.

Superior Carrier Gas Purity Plus Significant Savings

Sequentially connecting the High Capacity Heated Gas Purifier in-line with the OMI Indicating Purifier not only delivers the best possible protection for your GC columns and detectors, but also is the most cost-effective purification system you can buy. You no longer have to purchase costly certified gas to avoid critical carrier gas impurities – like O_2, H_2O, CO_2, and halogen and sulfur compounds – that can damage your GC columns and elicit detector noise. These sequentially connected purifiers remove impurities from inexpensive (99.995% pure) carrier gas.

Supelco researchers purify lower carrier gas grades with this purification system. After purification, our carrier gas actually contains lower concentrations of *chromatographically critical impurities* than most certified grades (Table 1).

You can also save money by using a Heated Gas Purifier to purify gas from a single cylinder for distribution to several GCs. A single gas line is connected from the cylinder of bulk gas to the heated purifier. After exiting the purifier, the gasline carrying the purified carrier gas is split to serve several GCs. If you want to further ensure carrier gas purity, connect an OMI Indicating Purifier immediately before each GC for point-of-use purification. The OMI's clear glass body provides visual assurance of your carrier gas' quality.

Table 1. Purity[1] of Gas from Supelco Purifiers Exceeds That of Certified Gas

	Typical Carrier Gas Impurity Levels (ppm)[2]		
Component	Certified Gas (99.9995%)	High Purity Gas (99.995%)	Supelco Purified Gas
Oxygen	1	3	0.01
Nitrogen	1	5	5
Water	0.5	1.5	0.01
Carbon Dioxide	0.1	0.5	0.06
THC (as CH_4)	0.5	1	1
Neon	1.0	1	1
Argon	0.5	0.5	0.5
Total Impurities	4.6	12.5	7.58
(excluding inerts[3])	1.6	5.0	0.08

1 Relevant to chromatographically critical impurities.

2 *Column 1* compiled from various specialty gas manufacturers' catalog specifications; *Column 2* from personal communication with industry experts; *Column 3* provided by Hercules, Incorporated, Wilmington, DE.

3 Inerts include nitrogen, argon, neon.

Ordering Information:

Description	Cat. No.
Supelcarb HC Hydrocarbon Trap	
120cc, 1/8" fittings	**24448**
120cc, 1/4" fittings	**24449**
750cc, 1/4" fittings	**24564**
750cc, 1/2" fittings	**24565**
"S" trap, 1/8" fittings	**503134**
Supelcarb refill, 300cc	**24566**
Supelpure-HC Hydrocarbon Trap	
120cc, 1/8" fittings	**22445-U**
120cc, 1/4" fittings	**22446**
750cc, 1/4" fittings	**24518**
750cc, 1/2" fittings	**24519**
"S" trap, 1/8" fittings	**503142**
Supelpure charcoal refill, 400cc	**22451**
Molecular Sieve 5A Trap	
200cc, 1/8" fittings	**20619**
200cc, 1/4" fittings	**20618**
750cc, 1/4" fittings	**23991**
750cc, 1/2" fittings	**23992**
"S" trap, 1/8" fittings	**503118**
Molecular Sieve 5A refill, 1/2 lb (0.22kg)	**20298**
Economy moisture-removing trap (for air service)	
400cc, 1/8" fittings	**23987**
400cc, 1/4" fittings	**23988**
Molecular Sieve 13X/4A refill, 1 pint (~475cc)	**23989**
OMI-2 Purifier (tube only)*	**23906**
OMI-2 holder	**23921**
OMI-4 Purifier (tube only)*	**23909**
OMI-4 Holder	**23926**
High Capacity Gas Purifier	
110VAC, 1/8" fittings (oven and purifier tube)	**23800-U**
110 VAC, 1/4" fittings (oven and purifier tube)	**23802**
220VAC, 1/8" fittings (oven and purifier tube)	**23801**
220 VAC, 1/4" fittings (oven and purifier tube)	**23803**
Supelpure-O Trap	
1/8" fittings, 120cc	**22449**
1/4" fittings, 120cc	**22450-U**
1/4" fittings, 750cc	**503088**
1/2" fittings, 750cc	**503096**
"S" trap, 1/8" fittings	**503126**
Mounting clips	
For 120cc traps	**23993**
For 200cc traps	**503231**
For 400cc traps	**23990**
For 750cc traps	**24983**
For "S" traps	**502901**
Zero Air Generator	
Model 1000 (100-125 VAC, 0-1000cc/min)	**22824**
Model 1001 (200-250 VAC, 0-1000cc/min)	**22830-U**
Model 2500 (100-125 VAC, 0-2500cc/min)	**22812**
Model 2501 (200-250 VAC, 0-2500cc/min)	**22814**

* First time users must order both OMI-2 holder and purifier tube.

For more information, or current prices, contact your nearest Supelco subsidiary listed below. To obtain further contact information, visit our website (www.sigma-aldrich.com), see the Supelco catalog, or contact Supelco, Bellefonte, PA 16823-0048 USA.

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