

The Importance of Pure Air for Maximum FID Performance

Have you ever asked yourself why your baseline drifts when using a flame ionization detector (FID)? Do you have difficulty integrating peaks because your baseline isn't flat? While you may have chosen the best grade of carrier gas and have installed purifiers to guarantee purity, there are special considerations for air that feeds the FID.

Determining air purity may not be straightforward. Gas purity is usually expressed in terms of "nines". For example, a cylinder may be referred to as 99.999%, or "5 nines" pure. These values are stated in terms of contaminants measured subtracted from 100. Unfortunately, not all contaminants are measured. Methane, for example, is commonly ignored in cylinder air. If you look closely, the manufacturer's purity certificate will often state that methane is not assayed.

The Problem

Conventional gas purifiers do not remove methane from air. A catalytic purifier employed in a zero air generator is needed to remove methane. Within the gas industry, the "zero" term ("zero air" for example) means that all hydrocarbons, including methane, are reduced. Residual methane can cause drifting baselines (sinusoidal effect), limit GC sensitivity, and cause peak integration problems. Because of this, zero air is critical for optimum performance of your FID.

Gas cylinders, house systems and laboratory compressors are all acceptable sources of air but each needs special consideration to supply air that is both methane and hydrocarbon free. But what is the best approach to removing hydrocarbons, including methane, from air?

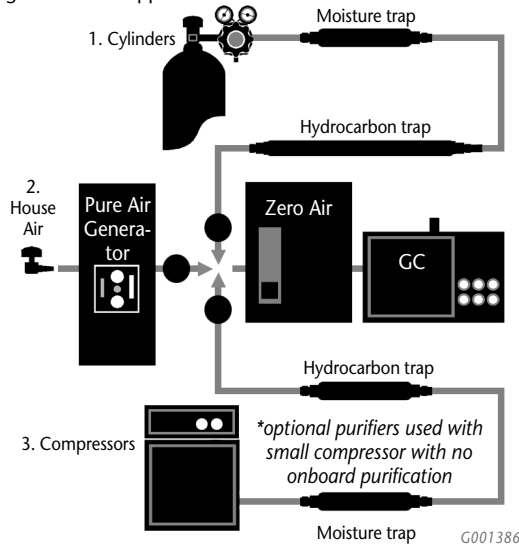
The Approach

Cylinders - The use of cylinders is ideal because the purity of the air is largely known. With cylinder air, in-line moisture and hydrocarbon purifiers such as the Supelco molecular sieve water trap and Supelcarb hydrocarbon trap must be installed to insure that the maximum cylinder impurities are below 100ppm for moisture and hydrocarbons, respectively. Next, add a zero air generator (Figure 1). Be sure to match the capacities of your purifiers with your expected flow requirements. Exceeding the flow capacity of a purifier will allow contaminants to break through.

A further advantage of zero air generators is in its cost savings. What many GC users do not realize is that a zero air generator used in conjunction with conventional purifiers yields the highest attainable purity air even from low-grade cylinder air! Because expensive ultra high-purity cylinder air is not required, significant cost savings are achieved.

GC
issue

Figure 1. Three Approaches to Pure Air



House Air - With proper precautions, house air is a good alternative to cylinder air. Most house air systems utilize oil-sealed compressors. While new house air systems provide low hydrocarbon and moisture emissions, older systems are more likely to pass oil vapors. Often, the quality, flow capacity, or available pressure of house air is unknown. Even if the house air system has moisture and hydrocarbon traps installed, this does not guarantee that all of the contaminants have been eliminated. Often, contaminants will accumulate in the lines over time. The solution is to install a high flow Pure Air Generator on your house system. These automatic cleaning and switching generators (purifiers) remove contaminants that appear seasonally with ambient temperature changes and safeguard your zero air generators. They have flow capacities ranging from 85L/min up to 366L/min.

Compressors - In some situations, the best solution is to install a non oil-sealed air compressor such as the Jun-Air oilless air compressor in the lab. Lab compressors eliminate your dependence on the scheduled plant maintenance of house systems and provide a dedicated system with full control of all aspects of the gas purity. Typical payback for a compressor is less than one year. These high flow compressors (42 – 100 L/min) come with moisture and hydrocarbon-trapping systems that meet the input specifications for zero air purity. Smaller units may not contain built in purifiers and may lack other features such as sound cabinets. All compressors should have a means to dampen compressor pressure surges.

(continued on page 4)

CONTENTS

Page 1:

- The Importance of Pure Air for Maximum FID Performance

Page 2:

- New Products
Gas Generation, Purification, and Delivery
- Sample Preparation and Introduction
- Fittings and Accessories
- Columns

Page 3:

- New Applications
- Seminars
- New Literature
- GC Performance Tip
Mass #100 Isn't the Column

Page 4:

- The Importance of Pure Air...(contd.)
- Case Study 3
A Drifting Baseline

SUPELCO
a member of the Sigma-Aldrich family

NEW PRODUCTS

Gas Generation, Purification and Delivery

domnick hunter Pure Air Generator



E000774

domnick hunter Pure Air Generator

Nitrox UHP pure air generators remove water, dirt, and oil from air using reliable pressure swing adsorption technology. Two beds of high performance desiccant alternate between moisture removal mode and regeneration mode, for continuous delivery of air at specified purity levels. High efficiency pre-desiccant and post desiccant filtration ensures total air quality.

Note: These units do not remove methane. Methane free air requires a zero air generator.

Model 070	27751-U
Model 140	27752-U
Model 300	27753-U

☎ For more information, request T499200.

domnick hunter Zero Air Generator



E000775

domnick hunter Zero Air Generator

Nitrox zero air generators employ a catalytic oxidation system to produce a continuous supply of air that is virtually free of hydrocarbons, including methane. Incoming compressed air is filtered to remove oil, water and particles and then passed through a heated catalyst that reduces hydrocarbon concentrations to below 0.1 ppm when the incoming air contains less than 10ppm hydrocarbons (as methane).

Model 10	27758-U
Model 35	27759-U

☎ For more information, request T499202.

Packard Model 9000 Hydrogen Generator



E000837

Packard Model 9000 Hydrogen Generator

Hydrogen generators are a safe and economic alternative to high-pressure gas cylinders. Supelco has added the Packard Model 9000, the newest member of the Packard generator family to our product line. This new model utilizes the same reliable Solid Polymer Electrolyte (SPE) technology as the higher capacity models of Packard hydrogen generators to produce 80 cc/min of 99.9999+% hydrogen. This affordable new model is an ideal source of hydrogen as fuel for one or two FID's.

Model 9000 hydrogen generator	27773-U
-------------------------------------	---------

☎ For more information, request T400021.

Sample Preparation and Introduction

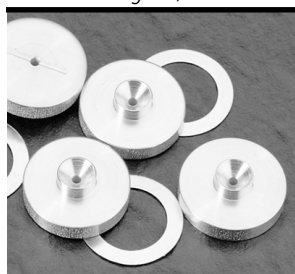
Inlet Seals for Agilent GC's

Low cost, replacement inlet seals for Agilent GC's from Supelco reduce the need for cleaning and reuse. Supelco metal selection yields a better inlet seal. Seals are available in stainless steel and gold plated versions. Precise, computerized machining reduces dimensional variation that can occur with other seals.

Stainless Steel HP Inlet Seals, Pk. of 2	23316-U
Stainless Steel HP Inlet Seals, Pk. of 10	23317-U
Gold Plated HP Inlet Seals, Pk. of 2	23318-U
Gold Plated HP Inlet Seals, Pk. of 10	23319-U

☎ For more information, request T400006.

Inlet Seals for Agilent/HP GC's

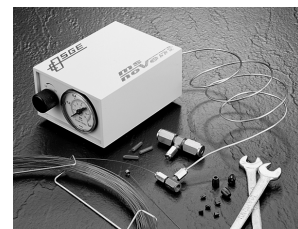


P000752a

Fittings and Accessories

ms-NoVent

A new time saving accessory for GC/MS systems, the ms-NoVent allows capillary users to begin using the mass spec within minutes after changing columns. The ms-NoVent supplies the mass spec with carrier gas during column change, eliminating system pump down. It consists of a pressure switching valve, fused silica restrictor, and an external control module. Installation in Agilent, Varian, Shimadzu, and other systems is typically less than 30 minutes.



P000699

ms-NoVent System

Restrictors for ms-NoVent System

Restrictor for HP5970, Qty. 2

Restrictor for HP 5971/5972, Qty. 2

Restrictor for HP 5973, Qty. 2

Restrictor for Varian Saturn, Qty. 2

Restrictor for Shimadzu 5000/5050, Qty. 2

☎ For more information, request T400005.

Septum Insertion Tool

Change a septum quickly without cooling the injector thanks to Supelco's new patented septum insertion tool. This tool installs a septum into any GC injection port that uses 9.5 - 11mm diameter septa. The tool compresses the septa and makes insertion into the injector easier. It reduces the potential for burned fingers and contamination from finger oils or other sources.

Septum Inserter

☎ For more information, request T400004.



P000258

Columns

Capillary Columns for Agilent 6850

Supelco makes it easy to purchase off-the-shelf capillary GC columns for the Agilent 6850! By referencing order code PRO100060, you can purchase any stock or custom Supelco capillary column wound on an authentic Agilent Technologies 6850 cage. To order, simply provide order code PRO100060 plus the stock capillary item number or custom column information. Supelco will coil the column onto a 6850 cage and ship the column within 24 hours.

☎ For more information, request T400051.

All literature mentioned in this issue can be obtained from the website, www.sigma-aldrich.com/TheReporter, by completing the Literature Request section on the reply card, or by calling our Technical Service Department.

NEW APPLICATIONS

GC/MS Analysis of Impurities in Denatured Ethanol

SDA is the most common form of denatured ethyl alcohol. It is also a solvent commonly used in industrial applications. Organic impurities can affect the product in which SDA is used. This application illustrates analysis of 19 impurities commonly found in SDA-3A, which is ethanol denatured with 5% methanol. GC/MS was used for the positive identification of contaminants. The application is unusual in that special precautions were taken to protect the MSD during the analysis.

For more information, request T300163.

Analysis of Fusel Oils in Alcoholic Beverages

Alcoholic beverage and coffee producers regularly analyze for fusel oils resulting from the fermentation and/or aging process. This separation has traditionally been run using packed column GC. In this application, an SPB-20 column is used to resolve twelve different contaminants from a rum stream at PPM levels. The application illustrates separation of the critical iso-amyl and active-amyl alcohol peaks. Total analysis time is less than 25 minutes.

For more information, request T300164.

SEMINARS

Analysis of Flavors and Off-Flavors in Foods and Beverages Using SPME

SPME is a convenient, solventless extraction technique that can be used to extract analytes from both liquid and solid matrices. The use of SPME for the analysis of flavors and off-flavors in food and beverages is important. In this presentation, sample types such as non-alcoholic and alcoholic beverages, candy, and fruits are analyzed for flavor composition. The detection of off-flavors from rancid oils and fats and methods for quantifying pyrazines in peanut butter and caffeine in coffee are presented. This poster was recently presented at the International Capillary Symposium in Riva del Garda, Italy.

For more information, request T400133.

Optimization of Extraction Conditions and Selection of SPME Fibers for VOA and Semi-VOA Analytes

The selection of an appropriate SPME fiber for an application can be a difficult process. Two experiments were performed. In the first experiment, volatile analytes of similar size from a variety of analyte classes were extracted from water. The second study is a repeat of this work using semi-volatile analytes. Results are discussed from evaluation of nine different SPME fibers on the extraction of fifteen semi-volatile analytes. The data illustrate the relationship of fiber coating thickness and polarity to analyte size and polarity. The differences between adsorbent and absorbent type fibers are explored. This poster was recently presented at the International Capillary Symposium in Riva del Garda, Italy.

For more information, request T400134.

NEW LITERATURE

Gas Purification Guide Updated

Selecting the proper purifiers for a GC system begins with determining the contaminants to be removed, the levels to which they must be reduced, and the flow and pressure needs of the system. The best purifier system includes multiple purifiers that help protect each other while protecting columns and detectors. This updated bulletin includes information needed to select suitable purifiers for carrier gas, and for air and hydrogen used as fuel gases.

For more information, request T197918.

SPME Application Guide Update

The SPME Applications Guide is a bibliographic resource of more than 600 published technical articles about solid phase microextraction. With the continued growth of SPME, and its expansion into environmental, food, forensic, and other fields, we recognize the need for an organized bibliography. This updated guide should serve a useful purpose in your SPME research and analyses.

For more information, request T199925.

Updated Gas Management Systems for GC

The information in this bulletin will help you to use your gas management resources wisely and obtain efficient performance from one, several, or many gas chromatographs. This updated bulletin includes installation information and diagrams specific for 1, 2-4, or 5-20 gas chromatograph systems. Common installations are illustrated. The bulletin discusses how to choose, clean, and connect tubing, the comparative merits of gas cylinders and generators, and how to attain suitable levels of gas purity. We recommend you read this updated bulletin along with your instrument manual before installing a GC(s).

For more information, request T196898.

Supelco Petroleum Guide Updated

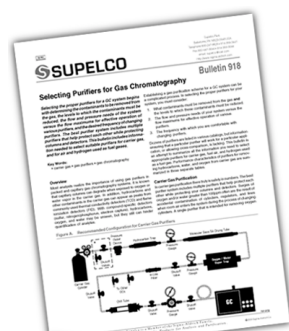
This 48-page guide contains information about Supelco products and technology for separating hydrocarbons by chromatographic methods. The updated guide illustrates nearly one hundred different hydrocarbon separations including PONA, PIANO, and SIMDIS applications. Multiple capillary and HPLC products are included to help with your analytical hydrocarbon needs. The guide is recently updated to reflect current products and applications available at Supelco.

For more information, request T100858.

GC PERFORMANCE TIP

Mass #100 Isn't the Column

Many GC/MS chromatographers are used to seeing mass #207 or #73 background from methyl silicone columns. Sometimes you see mass #100. Where does this come from? Graphite/Vespel ferrules used to connect the column to the MSD interface are rich sources of mass #100 and may cause background if the ferrule is incorrectly installed or if the wrong ferrule is used. If you see background, check the column installation and the ferrule used at the interface.



Len Sidisky - R&D Manager,
Gas Separations Business Unit

Trademarks and Registered Trademarks:

Agilent - Agilent Technologies, Inc.
domnick hunter, **Nitrox** - domnick hunter Limited
Jun-Air - Jun Air A/S
Packard - Hewlett Packard Company
Shimadzu - Shimadzu Corporation
Supelcarb - Sigma Aldrich
Varian - Varian Associates

Patents:

SPME - Technology licensed exclusively to Supelco. US patent #5,691,206; European patent #523092.

The Importance of Pure Air...

(continued from page 1)

When thinking about a compressor, consider site location, total flow, and pressure requirements. Since compressors vibrate, they should be located on the floor away from hydrocarbon sources. Compressors require a water vent since they can condense gallons of water per day from the air. Although most compressors are quiet (<60 decibels), locate them away from desks to avoid distractions. Moisture and hydrocarbon traps are required with compressors that don't have them. Remember that a zero air generator is required to eliminate ppm levels of methane normally present in air.

When sizing a compressor, be sure to include all your lab flow demands and allow a little extra. If supplying air for permeation type nitrogen generators, for example, you may need as much as 66L/min of air per 1L/min of nitrogen generated.

Conclusion

Purified air, whether the source is from cylinder, house, or compressor, is less expensive when obtained using purifiers

and generators. The long term cost savings of using low-grade cylinder air are significant and should be considered when designing your air system. Any of the three air sources discussed, when passed through purifiers and a zero air generator, will provide air containing less than 1ppm of all hydrocarbons, including methane, and will result in increased sensitivity and smoother baselines from your FID.

If you need help selecting the correct equipment for your specific application, give our Technical Service department a call. Also, consider the following literature available from Supelco.

Recommended Reading:

Bulletin T196898 for information on installing cylinders, tubing, regulators, compressors, and gas generators

Bulletin T197918 for information on purifier capacity and the cost effectiveness of purifiers versus high quality gases

Bulletin T198921 for the cost effectiveness of using generators and purifiers in place of high purity cylinder gases

CASE STUDY 3

A Drifting Baseline

Supelco chemists were generating fatty acid methyl ester (FAME) chromatograms recently and were having difficulty with baseline drift during the analyses. Randomly, the baseline drifted during otherwise acceptable chromatography. The chemists could not explain the drift. Figure 2 illustrates the chromatography observed by the chemists.

The Investigation – Reviewing chromatograms, the chemists observed that the FID baseline drifted as high as 20pA. A normal FID baseline is smooth and at a level of about 5pA. This pointed to a contaminated detector. However, after cleaning the high, drifting baseline remained. Chemists investigated the house gas used to supply the GCs to learn that deliveries or other changes to the bulk gas system had not occurred.

They then rechecked the basics, inspecting every facet of the gas purification system. Gases plumbed to the GC system were scrubbed using the appropriate high capacity and indicating purifiers. Traps had been changed only a few weeks earlier during routine system maintenance, and acceptable chromatography was generated less than one day earlier. The drifting baseline suggested that contamination was randomly being introduced to the detector during analysis. Someone then recalled that a similar problem occurred during an unusually hot June several years earlier before the zero air generator was installed. Pursuing this concept, chemists decided to examine the zero air generator more closely.

The Solution – After examining the system, the chemists discovered that the zero air generator was installed on a

different electrical circuit than the rest of the system. Intermittent electrical maintenance was being performed on the circuit supplying power to the unit, and occasional power loss was interrupting normal generator operation. The problem was corrected and after a short warm up, the FID baseline stabilized. Figure 3 illustrates the results once power was fully restored to the device.

For more information, request T198921.

Figure 2. Poor Baseline – Zero Air Generator not Working

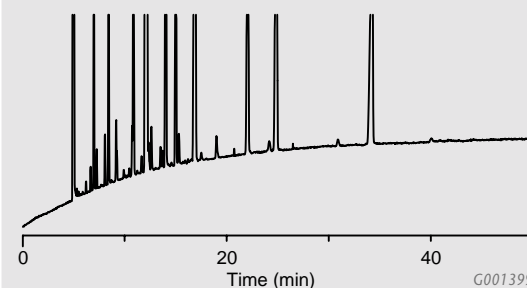
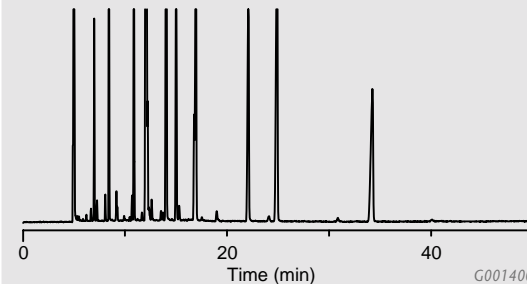


Figure 3. Stable Baseline – Zero Air Generator Working Correctly



SUPELCO • Supelco Park, Bellefonte, PA 16823-0048 • 800-247-6628 • www.sigma-aldrich.com



The
SIGMA-ALDRICH
Family

SIGMA
Biochemicals and
Reagents for Life
Science Research

ALDRICH
Organics and
Inorganics for
Chemical Synthesis

Fluka
Specialty Chemicals
and Analytical
Reagents for Research

SUPELCO
Chromatography
Products for Analysis
and Purification

Riedel-deHaën
Laboratory Chemicals
and Reagents for
Research and Analysis

© 2000 Sigma-Aldrich Co. Printed in USA. Supelco brand products are sold through Sigma-Aldrich, Inc. Sigma-Aldrich, Inc. warrants that its products conform to the information contained in this and other Sigma-Aldrich publications. Purchaser must determine the suitability of the product(s) for their particular use. Additional terms and conditions may apply. Please see reverse side of the invoice or packing slip.