

PAMSGRAM

PLEASE DELIVER TO ALL RECIPIENTS IN YOUR OFFICE

Volume 11
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The PAMSGRAM is a technical information notice for Federal, State and Local air pollution control agencies

PAMSGRAM Distribution

Notice: Future editions of the **PAMSGRAM** will be posted on the Ambient Monitoring Technology Information Center (AMTIC) home page and the Enhanced Ozone Monitoring (PAMS) Web Site at www.epa.gov/ttn/amtic/pamsgram.html, rather than being FAXED. EPA will notify interested parties via e-mail when a **PAMSGRAM** is posted and provide an electronic copy. If you wish to receive an e-mail notice and the electronic **PAMSGRAM**, please send a message to gerald.nash@epa.gov. For the latest on PAMS, check out the PAMS Web Site at www.epa.gov/oar/oaqps/pams/ or the new Enhanced Ozone & Precursor Monitoring interactive site at <http://capita.wustl.edu/EnhancedOzone/>.

Retention Time Cylinders for the PAMS 1998 Season

As you probably know, the retention time cylinders purchased for the 1998 PAMS season are just being delivered to the regional, state and local agencies. The cylinder purchase was delayed due to administrative and logistical issues. In any event, the cylinders are arriving at the end of the PAMS season. When the new 1998 retention time cylinder is received, EPA recommends that users analyze the cylinder upon receipt as a second-source quality control check to verify their current VOC instrumentation calibration and target peak identifications.

The compounds contained in the new retention time cylinder are those listed in **PAMSGRAM**, Volume 9, issued on March 18, 1998, with **some exceptions**. Two non-target compounds were inadvertently omitted from the 1998 cylinder mixture. These are 1-hexene and dodecane, which were added in 1997 to serve as retention time markers for C₆ and C₁₂ compounds. The cylinder contents are virtually identical to that of the 1996 mixture and does include 2-methyl-1-hexene (a C₆ compound known to be unstable and removed from the 1998 PAMS target list). EPA will be soliciting input through the EPA Regions and is currently investigating its options regarding the resolution of this issue.

An additional note of importance, letters were recently sent to those of you retaining **old retention time cylinders** which carry a monthly demurrage (rental). The cylinders with demurrage were purchased prior to 1997 (1996 and older). The 1997 and

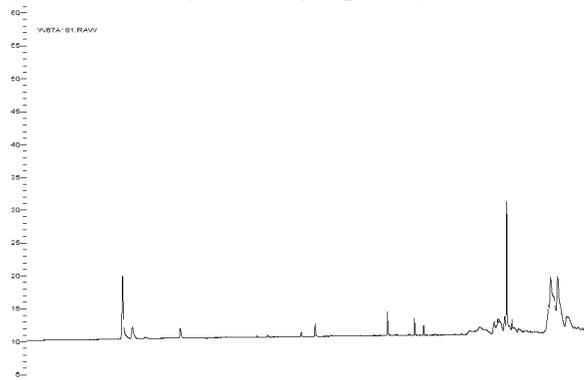
1998 cylinders were purchased outright and do not carry a monthly demurrage; therefore, they can be kept indefinitely. If you wish to purchase the old cylinders, please contact Julie Swift at 919-468-7924.

To return the old cylinders, please ship them to:

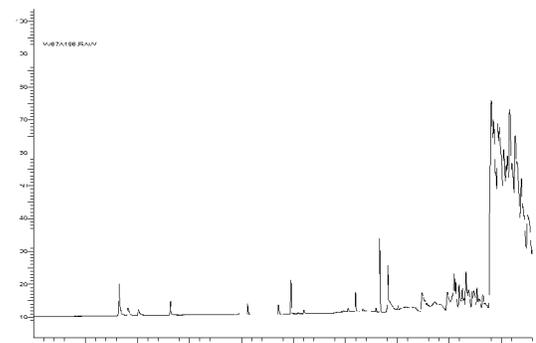
Scott Specialty Gases, Inc.
1750 East Club Blvd.
Durham, NC 27704
919-220-0803

Automated GC Insights

Recently, the Connecticut DEP noticed a “creeping sickness” in their air monitoring systems. Insidious at first, it was just an elevated baseline at the high end of the PLOT column run. Not enough to disable integration but just plain *ugly*.



Then it really got bad, as you can see from the following



Turbochrom® chromatogram. What could it be? Well, we have started to narrow it down but since the systems are now running for the season and collecting data we cannot do more experimental work until the Fall. It seems that this phenomenon may have something to do with polar species such as alcohols, aldehydes and ketones.

In gas chromatography we try to use a column that has a similar stationary phase to the type of compound that we're trying to separate. Thus a BP-1 column is good for non-polar hydrocarbons because its phase is distinctly non-polar. However the PLOT column is highly specialized because it must retain and separate the C₂ hydrocarbons (ethane, ethylene and acetylene) using "polarity". The only distinction between the C₂ hydrocarbons is the degree of saturation, which affects the polarity of the molecule. The special salt (sodium sulphate) used in the preparation of the phase provides the polar surface which retains and separates the C₂ hydrocarbons at ambient temperatures.

But this same high degree of polarity which is great for acetylene is really bad for moisture and alcohols. Water is usually not a problem because we get rid of it with a Nafion® dryer. Most incoming polar compounds are not a problem either because the dryer takes them out at normal concentration levels. Now try to imagine what happens when a very large amount of a polar solvent accidentally gets onto the PLOT column. Catastrophe! The solvent binds so tightly to the phase that it cannot move along very quickly, and this is exacerbated by the short (6 minute) time at the highest temperature of 200°C. So the "peak" moves very slowly until about 4 to 12 hours later there is a huge upset as it starts to elute and be detected. How does the polar compound get onto the PLOT column? We're not totally sure, but it seems likely that Leak Testing is one of the scenarios. Therefore, effective immediately, it is recommended that alcohol solutions and other polar solvents be removed from leak-testing procedures. This includes soap-solutions and other doubtful materials, including the old favorite - methanol and water. Use a normal hydrocarbon instead, such as cyclopentane or isopentane. You know that these materials will come off the PLOT column if they get onto it accidentally. Usually an electronic leak detector is helpful, but if you don't have one, try to use one of these fluids **bearing in mind that they are highly flammable**. Use good ventilation and sound judgement at all times. If you see anything like this on your own system, disable the Deans' switch immediately to prevent any more of the material from entering the PLOT column, and get some advice on how to proceed. One last note - watch out for dirty airstream humidifiers, tubing and other sources of potential bacterial action. Reservoirs used for humidification should be sterile to ensure that potentially damaging off-gases are not created by microorganisms. Sterilize the system annually.

How to Leak Test an ATD System

After major overhauls or if a leak is suspected you should always perform a leak-down pressure check, as follows:

- 1) Stop ATD and place in Standby Mode with Page 3 displayed showing MIN PSI value.
- 2) Remove PLOT and BP-1 columns from detectors and plug the ends by pushing the tips into a septum.
- 3) Set midpoint pressure to equal ATD MIN PSI actual value

(say 48 psi). Allow system to stabilize for 5 min.

- 4) Rapidly unscrew pressure regulator controls (CCW) on ATD and midpoint control until they are fully backed out.
- 5) Observe the MIN PSI actual pressure. It should not fall quickly (leak) but drop very slowly (no leak), about two to five psi over 5 minutes.
- 6) If a leak is observed, re-pressurize the system and use a leak detector. Check fittings using wrenches.
- 7) After the leak is fixed, trim 1 cm from each column end and re-insert into detectors. (Always trim the ends to remove possible plugs.) Restore pressure settings.

NOTE: If you have access to a millivolt recorder you can plot the transducer output pressure curve by monitoring PL-48 on the I/O board.

The graphite seals used on the cold trap can sometimes leak at high temperature during the desorb cycle, yet pass leak-test at low temperatures. Please be aware of this. Also, the glass-lined stainless-steel tubes into the Valco valve are sometimes (accidentally) not fully inserted. This leads to a similar high-temperature leak.

Disclaimer: Mention of trade names or commercial products in this EPA PAMSGRAM are not intended to constitute endorsement or recommendation for use.



If you have an experience (relating to air monitoring please) that you'd like to share, please send an email to Joann Rice at rice.joann@epa.gov. Don't be shy! Or if you have a special request for information you'd like to see, please let us know.