

Chromatography Corner

this issue

Advanced Cooling System P.1
PDHID Analysis P.2
Chromatography Tips & Tricks P.3
Events Calendar P.4

upcoming events

- **February 17:** Free Tedlar Bag Autosampler Webinar
Time: 9:00 am MST
- **March 31:** Free Automator Webinar
Time: 9:00 am MST

To register for one of Wasson-ECE's webinars visit:
www.wasson-ece.com/events
or call (970)221-9179

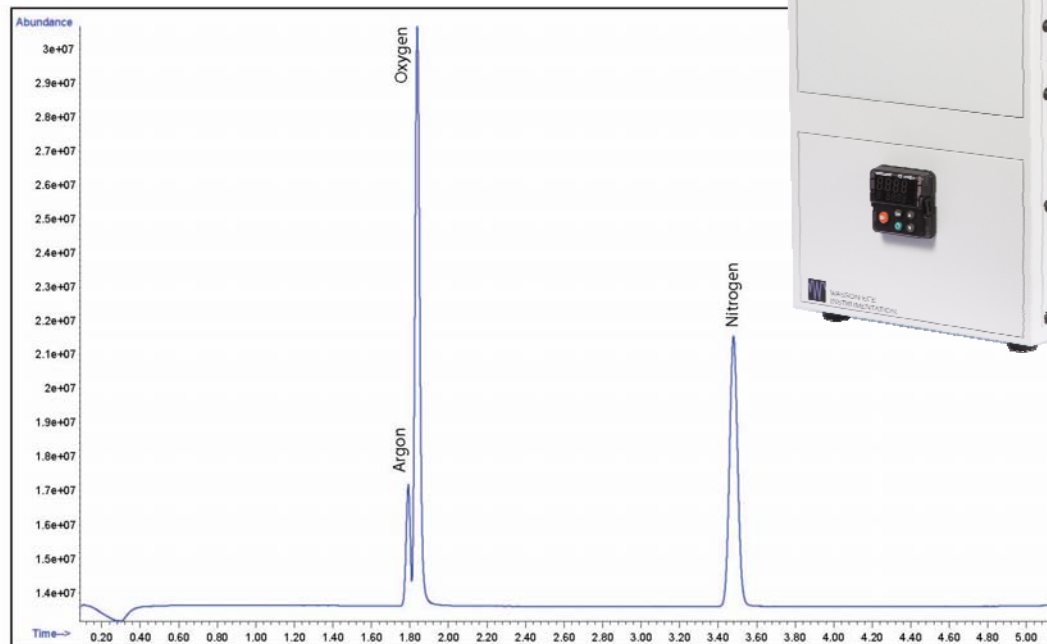
ISSUE 14 February 2010

Introducing the Advanced Cooling System

The new Advanced Cooling System (ACS) from Wasson-ECE Instrumentation provides an alternative cooling method for low temperature gas chromatography applications. The isothermal system is designed to integrate one column into an auxiliary box that can be cooled to 0°C. This allows the user to use the primary GC oven for higher temperature analyses. By running the ACS and the GC primary oven in tandem, low temperature applications can be run faster, saving valuable analysis time and increasing sample throughput.

The ACS offers an advantage for the lab that needs rapid throughput for low temperature gas chromatography applications without using liquid cryogen. The temperature is defined by the user and can range from ambient to 0°C. The ACS works well for light gas separations including argon/oxygen, sulfur dioxide/carbonyl sulfide, and nitrogen/oxygen on porous polymer columns.

Figure 1: Five minute analysis of Ar, O₂ and N₂ using the Advanced Cooling System and



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Trace Impurities in Perfluoropropane and Perfluorotetrahydrofuran by PDHID

Perfluoropropane (C_3F_8) and perfluorotetrahydrofuran (C_4F_8O) are non-flammable greenhouse gases. Greenhouse gases absorb and emit radiation within the thermal infrared range. This process is the fundamental cause of the greenhouse effect. They can be produced either by electrochemical fluorination or by the use of a cobalt fluoride. In the electronics industry, C_3F_8 and C_4F_8O are mixed with oxygen and used as a plasma etching material for SiO_2 layers in semiconductor applications since oxides are selectively etched versus their metal substrates. Because of their use in the semiconductor industry, C_3F_8 and C_4F_8O must be very pure and impurities must be quantified to low part per million (ppm) levels.

For the analysis of trace impurities in C_3F_8 and C_4F_8O , Wasson-ECE configured an Agilent GC with a pulse discharge helium ionization detector (PDHID).

The principle mechanism for the PDHID uses helium as its ionization source. Helium is introduced at the top of the detector, while the sample in the carrier gas is introduced from the bottom. Components are ionized by photons from the helium discharge. Resulting electrons are focused toward a collector electrode by two bias electrodes.

Introducing the sample and discharge gas at opposite ends minimizes electrode contamination by the components eluting from the analytical column.

The PDHID was a good detector choice for this analysis because it has a universal response to organic and inorganic compounds, with the exception of neon, which has an ionization potential higher than helium. Response is linear over five orders of magnitude, with LDLs in the low ppm range.

Argon/oxygen composite, nitrogen, methane, and carbon dioxide were quantified to 5 ppm. While hydrogen and carbon monoxide were quantified to 1 ppm. Samples were vaporized prior to injection with a total analysis time of 15 minutes.

By utilizing a very sensitive and universal detector like the PDHID, impurities in C_3F_8 and C_4F_8O were able to be detected and quantified to low ppm levels.

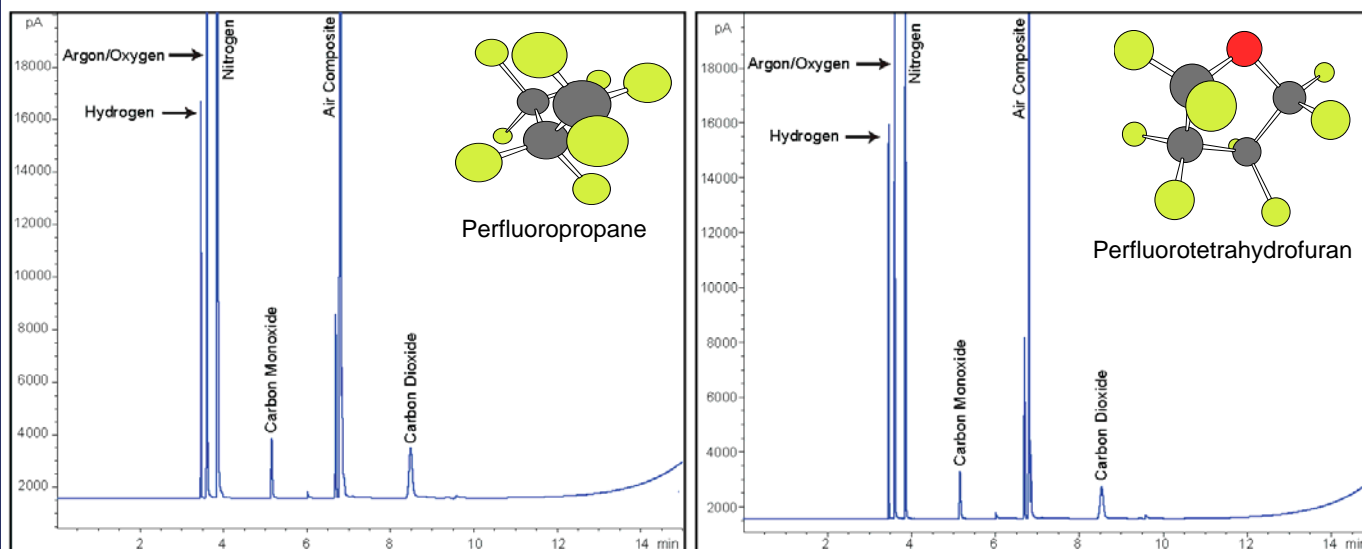


Figure 2 and 3: Analysis of impurities at ppm levels in perfluoropropane and perfluorotetrahydrofuran by PDHID.

Chromatography Tips and Tricks

As previously discussed in the January 2010 Issue of *Chromatography Corner*, the most common causes of GC column stationary phase degradation are:

- Column contamination from non-volatile and semi-volatile sample residues
- Oxygen damage at elevated temperatures
- Thermal damage by exceeding maximum temperature limits
- Chemical damage from inorganic bases, inorganic acids, and salts

Column contamination was discussed in a previous issue. This month oxygen, thermal, and chemical damage will be covered.

A leak in the carrier gas flow path (e.g. gas lines, fittings and injector) is the most common source of oxygen exposure. While these leaks won't cause damage at ambient temperatures, severe stationary phase degradation appears at elevated temperatures with oxygen concentration as low as 10 ppm. Symptoms include premature onset of column bleed, peak tailing for active compounds, and loss of resolution. The best way to avoid these types of contaminations is to do the following steps:

1. Use ultra high purity carrier gas.
2. Install a moisture and oxygen trap in all carrier gas lines.
3. Perform periodic leak checks of all gas lines and regulators.
4. Change septa regularly.
5. Change gas cylinders before they are empty.



Exceeding a column's upper temperature limit for a prolonged period of time or heating a column without carrier gas flow even slightly above ambient temperatures can result in degradation of the stationary phase. As polarity increases the temperature at which this damage occurs gets lower. Symptoms of thermal damage are similar to those of oxidation and include poor peak shape for active compounds, loss of retention, and elevated background signal. You can maximize the life of your column by setting the maximum GC oven temperature at or below the manufacturer's specified temperature limit. If the column is already damaged follow these steps. Condition it for 8-16 hours at an appropriate isothermal temperature. Remove 10-15 cm from the detector end and reinstall the column. You can continue to use the column, although the lifetime and performance may be shortened.

Inorganic bases such as KOH, NaOH, and NH₄OH and inorganic acids such as HCl, H₂SO₄, H₃PO₄, and HNO₃ are particularly damaging to stationary phases, causing peak tailing, or broadening, adsorption of active compounds or rising baseline. Chemical damage can be minimized by using a guard column, but keep in mind that frequent trimming may be required. Chemical damage is usually limited to the front of the column, so trim 0.5-1 meter from the front of the column (5+ meters in severe cases) to eliminate most chemical chromatographic problems.

Additional questions? Contact our service department at (970)221-9179 or service@wasson-ece.com.

Wasson-ECE Instrumentation News

New for 2010!

Wasson-ECE will be taking our 2-day Basic GC Course on the road.

April 14-15: Houston, TX

June 16-17: Los Angeles, CA

August 11-12: Baton Rouge, LA

October 13-14: Martinez, CA

Cost: \$1000* per participant

*Register before 2/28/2010 and receive 20% off the 2-day Basic GC Course.

Sign-up at www.Wasson-ECE.com and click on the Education Center or call (970) 221-9179.



Events Calendar



Wasson-ECE Instrumentation

specializes in configuring and modifying new or existing Agilent Technologies gas chromatographs. Our systems are guaranteed, turn-key analytical solutions, with the installation, warranty and service plan on us. Contact us for your custom GC analysis needs and find out what a difference over 20 years of experience can make.

- February 17:** Free Tedlar Bag Autosampler Webinar
- March 31:** Free Automator Webinar
- April 14-15:** Basic GC 2-Day Course in Houston TX
- April 21:** Free Webinar on New Wasson-ECE Hardware TBD
- May 26:** Free Blender with Mass Flow Controller Webinar
- June 16-17:** Basic GC 2-Day Course in Los Angeles, CA
- June 23:** Free Fast ASTM D3606 Webinar
- July 21:** Free Ambient Air Concentrator Webinar
- August 11-12:** Basic GC 2-Day Course in Baton Rouge, LA
- August 25:** Free Webinar Covering a New Wasson-ECE GC Application TBD
- September 22:** Free Eclipse Webinar
- October 13-14:** Basic GC 2-Day Course in Martinez, CA
- October 20:** Free Webinar Covering a New Wasson-ECE GC Application TBD
- November 17:** Free Webinar on New Wasson-ECE Hardware TBD

Want a custom training course for your company? Need training at your site? Contact Wasson-ECE for your quote today at training@wasson-ece.com or call (970)221-9179.



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