Monthly Newsletter From Wasson-ECE Instrumentation:

Chromatography Corner

ISSUE 13 January 2010

this issue

upcoming events

- **January 20:** Free El Paso Stripper Webinar
  Time: 9:00 am MST
- **February 17:** Free Advanced Cooling System 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

Analysis of Noble Gases in Air by PDHID

Wasson-ECE configured an Agilent gas chromatograph (GC) with a pulse discharge helium ionization detector (PDHID) for the analysis of noble gases in air. Components identified during method development included: argon, oxygen, krypton, nitrogen, and xenon to part per million (ppm) levels.

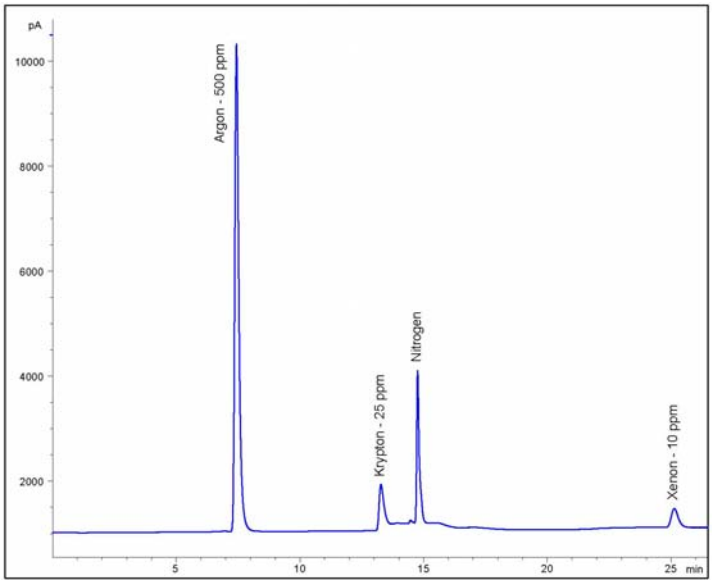
The detection of krypton was difficult due to the interference with the high levels of nitrogen in air. Liquid nitrogen cryogenic cooling was required to separate krypton from nitrogen as well as argon from oxygen.

This system utilized an O_2 scrubber system to remove oxygen from samples once they were injected so that trace argon could be detected in air. The system was configured with two O_2 scrubbers, for alternating between sample analysis and regeneration mode. After O_2 breakthrough was seen, the alternate scrubber was moved in line with a valve switch.

This design eliminated the need for downtime due to scrubber regeneration. Each scrubber system was able to accommodate more than 100 injections before requiring regeneration.

Two methods were created for the analysis of noble gases in air. Method 1 utilized the O_2 scrubber to separate and quantify argon. Method 2 bypassed the O_2 scrubber to allow oxygen to be detected by the PDHID. Each method had a runtime of approximately 30 minutes.

By using cryogenic cooling, an O_2 scrubber and PDHID, Wasson-ECE was able to quantify and separate noble gases in air samples.



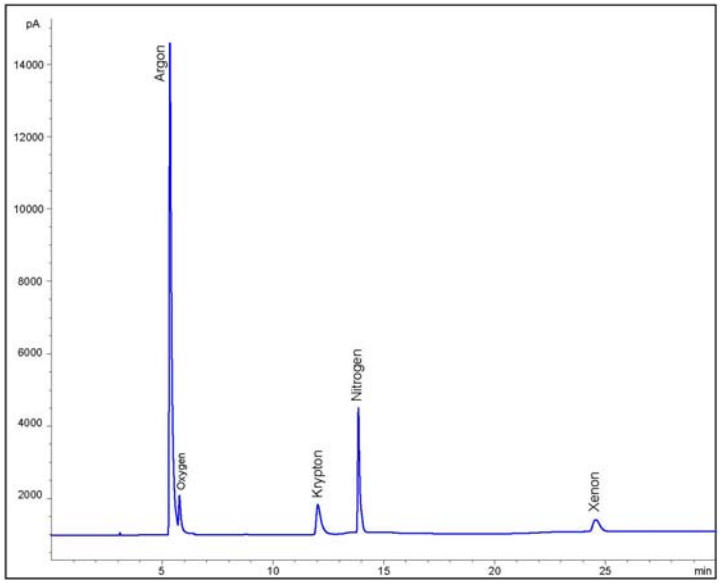


Figure 1: The O_2 scrubber is used to separate and quantify argon.
Figure 2: The O_2 scrubber is bypassed to quantify oxygen by PDHID.

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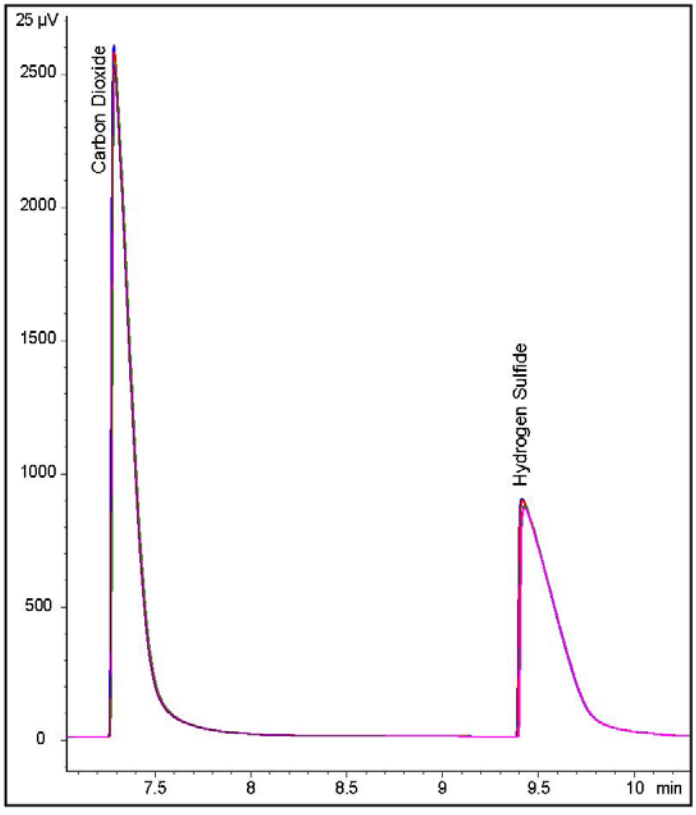
Analysis of H_2S and CO_2 in Amine Solution Using a Custom Sparge and Trap System

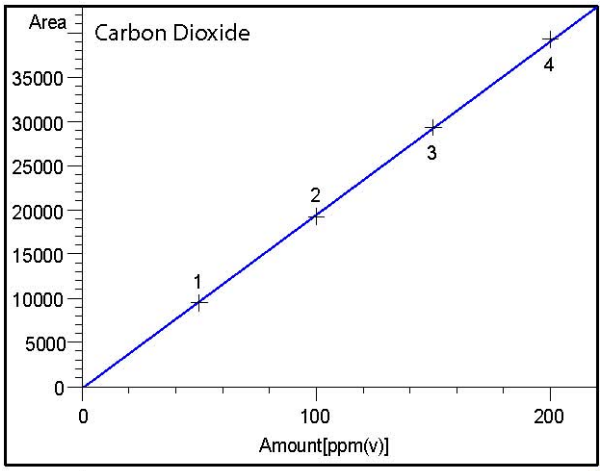
For the analysis of hydrogen sulfide (H_2S) and carbon dioxide (CO_2) in an amine solution, Wasson-ECE custom built a sparge and trap system to work in conjunction with a GC configured with a thermal conductivity detector (TCD). The methyldiethanolamine (MDEA) solution was used to scrub H_2S and CO_2 from natural gas. This process is known as gas sweetening because it results in products which no longer have the sour, foul odors of H_2S.

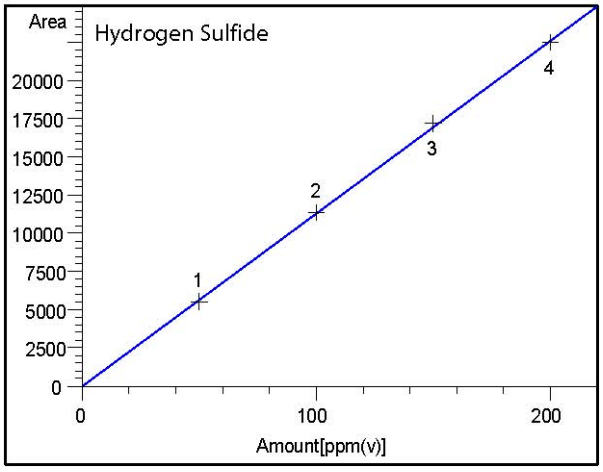
The samples were automatically diluted, and sulfuric acid added using a LEAP CTC auto-sampler. The sample was sparged with helium to remove the evolved gas from the solution. Prior to trapping, the sample was passed through a Nafion membrane drier to remove excess water. The evolved gas was then focused using an ambient adsorption trap and the analytes of interest were desorbed to the GC for quantification by TCD.

The system was able to detect H_2S and CO_2 gases without interference from the following trace gases that may be sparged from the sample: air, gas saturated with water vapor, C_1-C_7 hydrocarbons, BTX hydrocarbons, carbonyl sulfide, carbon disulfide, carbon monoxide, sulfur dioxide, hydrogen cyanide, mercaptans, and low molecular mass volatilized organic acids such as formic acid, hydrogen chloride, acetic acid, ammonia, alkanolamines, methanol, ethanol, acetone, and acetonitrile.

Sample load volumes could be varied to allow for flexibility in the analytical method. With a runtime of 20 minutes per injection, up to 30 samples can be run in a 24 hour period.







Figures 3 and 4: Chromatogram repeatability of a 150 ppm sample over 5 runs and linearity of the adsorbent trap for carbon dioxide and hydrogen sulfide when the load volume is varied.

Chromatography Tips and Tricks

If you want to maximize the life of your column, it is important to understand what causes column stationary phases to degrade and what can be done to minimize the effects.

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

While a decrease in capillary column performance can be attributed to these four causes, the primary cause of failure is contamination from residues in the injected sample. This article will focus on performance tips that minimize this type of damage. Tips for minimizing oxygen, thermal and chemical damage will be covered in the February 2010 Issue of *Chromatography Corner*.

Common symptoms of column contamination are high bleed, peak tailing, and reduced response to active analytes.

Non-volatile contaminants accumulate in the column, and therefore do not elute. The column becomes coated with residues that interfere with active solutes and cause tailing or peak size problems.

Accumulated semi-volatile contaminants might not elute for hours or possibly days. In addition to causing peak shape and size problems, semi-volatile residues are usually responsible for baseline instability and drift, as well as ghost peaks.

The following tips can be used to maximize the life of your capillary column and minimize column contamination.

1. **Check for residues.** Deposit about 20 µl of sample onto a microscope slide. Set the slide over a heated injection port until dry. If you can see any residue where the sample was, you may potentially be shortening the life of your column with damaging sample residues.
2. **Use a guard column.** Guard columns protect your analytical column from contamination. Sample residues are deposited in the guard column without coating the stationary phase of the analytical column. Guard columns are usually 5-10 meters to allow for substantial trimming before the entire guard column has to be replaced.
3. **Limit bake-out times.** High temperatures over long periods may convert some residues into insoluble materials that shorten column life. Limit bake-outs to 1-2 hours at the isothermal temperature limit of the column.

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

New for 2010 Wasson-ECE Training on the Road!

Wasson-ECE will be taking our 2-day Basic GC Course on the road. See below for scheduled dates and cities.

April 14-15: Houston, TX
June 16-17: Los Angeles, CA
August 11-12: Baton Rouge, LA
October 13-14: Martinez, CA

*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.

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.

Events Calendar

January 20: Free El Paso Stripper Webinar per EPA 40 CFR Parts 9 and 63
February 17: Free Tedlar Bag Autosampler Webinar
March 25: Customer Appreciation Night in Houston- *join us for a night of food and fun!*
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 D2887 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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