

# Chromatography Corner

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## upcoming events

- April 14-15: Basic GC Course  
Where: Houston, TX  
Cost: \$1000 per person
- April 21: Free Low Level Impurities in Permanent Gases Webinar  
Time: 9:00 am MST

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

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## Analysis of Trace Impurities in He, H<sub>2</sub>, and Ar

There are a wide variety of industrial applications that require uncontaminated helium (He), hydrogen (H<sub>2</sub>) and argon (Ar) gases including uses in the semiconductor and petroleum processing. Both of these processes require uncontaminated gases due to the pure and expensive nature of the final products.

Wasson-ECE configured an Agilent GC with a Valco pulsed discharge helium ionization detector and dual thermal conductivity detectors (PDHID/TCD/TCD) for the analysis of trace impurities in He, H<sub>2</sub>, and Ar.

Components analyzed on the PDHID included hydrogen, argon, oxygen, nitrogen, methane, carbon monoxide, and carbon dioxide. All components were analyzed to a lower detection limit (LDL) of 50 ppb, except hydrogen and carbon monoxide, which were 100 ppb.

Components analyzed on the TCDs included hydrogen (100 ppm), carbon dioxide (200 ppm), oxygen/argon composite (200 ppm), nitrogen (200 ppm), methane (200 ppm), and carbon monoxide (400 ppm).

Three different methods were created and could be used with each of the different matrix gases. The fast method analyzed low level impurities in the various gases by PDHID. The second method analyzed permanent gases by TCD and the third method analyzed hydrogen in only He and Ar.

By configuring a GC with a PDHID and dual TCDs a complete analysis for impurities in He, H<sub>2</sub>, and Ar could be done with an analysis time of less than 20 minutes for each gas matrix.

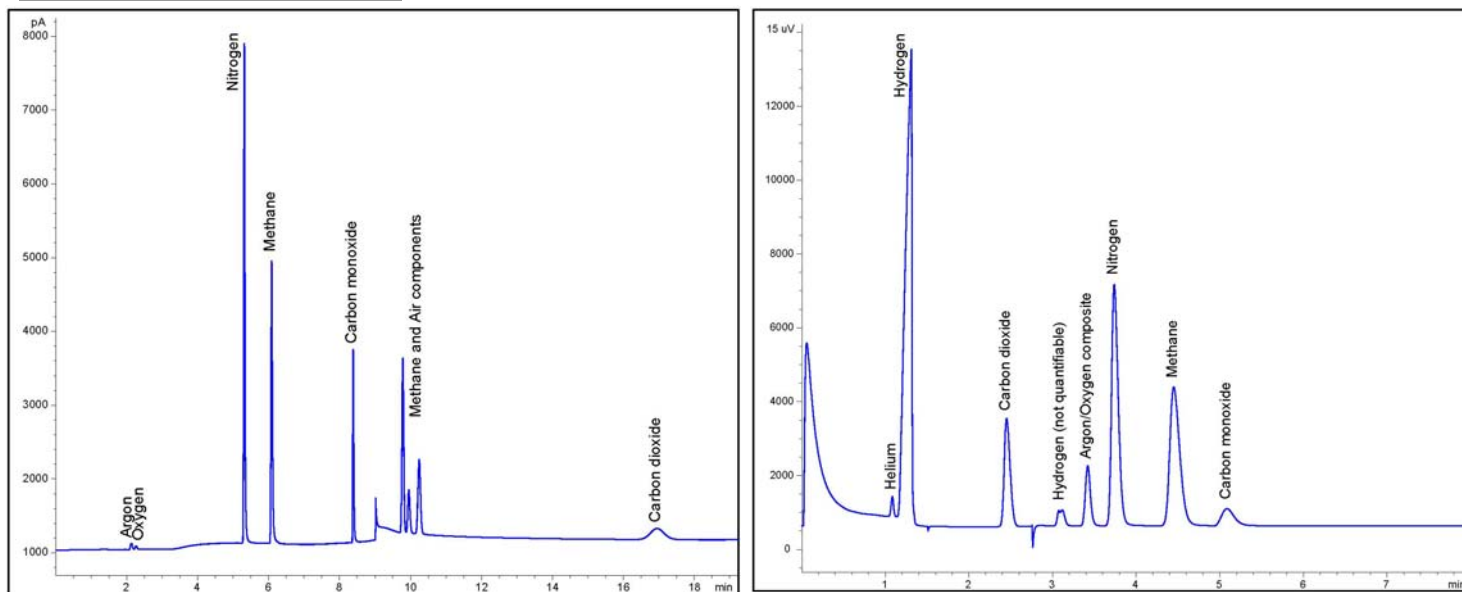


Figure 1: Analysis of trace impurities in helium by PDHID at ppm levels (left) and analysis of impurities in permanent gases by summation of dual Wasson TCDs (right).



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## Analysis of Cooling Tower Water in Compliance with TCEQ Chapter 115 Regulations

Wasson-ECE configured and tested an Agilent gas chromatograph (GC) to perform the analysis of cooling tower water in compliance with TCEQ chapter 115 regulations and the analysis of cooling tower water. The system utilizes flame ionization and photoionization detectors (FID/PID) to perform the complete analysis.

Components identified on the FID during method development included ethylene, propane, cyclopropane, propylene, acetylene, isobutane, propadiene, n-butane, trans-2-butene, 1-butene, isobutylene, cis-2-butene, neopentane, isopentane, methyl acetylene, n-pentane, and 1,3-butadiene. 3-methyl-1-butene also elutes from the column after 1,3-butadiene, but this peak is non-quantifiable. The lower detection limit was 0.01 ppmw for each component in the cooling water (using the TCEQ calculation). Components heavier than 1,3-butadiene were backflushed to vent. Components analyzed on the PID included ethylene, cyclopropane, propylene, acetylene, propadiene, trans-2-butene, 1-butene, isobutylene, cis-2-butene, methyl acetylene, and 1,3-butadiene which also had an LDL of 0.01 ppmw.

The PID allows for analysis of olefins while minimizing interference from paraffin signals. The PID and FID were linked in series with components eluting from the column to the in-line PID and passing from the PID to the FID.

This type of inline linking of the detectors is possible because as the PID performs non-destructible analysis.

The stand-alone concentration system is used in conjunction with the GC for the analysis of low level volatile organic carbon species (VOCs). Concentration factors of 100 to 500 times were achieved. The software interface allowed the user to enter a load volume between ten and 600 milliliters. An electronic mass flow controller regulated the load volume during sampling onto a sorbent trap, which was run at ambient temperatures.

The concentrator desorbs the sample directly to the GC initiating the start of the method in ChemStation. The concentration system then automatically checked for the ready signal and started the GC remotely at the time of injection. The concentrator included dry purge and bake-out routines to optimize the performance of the analysis.

When the air concentrator is used, certain compounds are not retained by the trapping material used. Compounds lighter than ethylene and linear compounds such as acetylene and propadiene are not well retained by this particular trap. Peaks representative of these compounds may appear in chromatograms, however, they are not quantifiable.

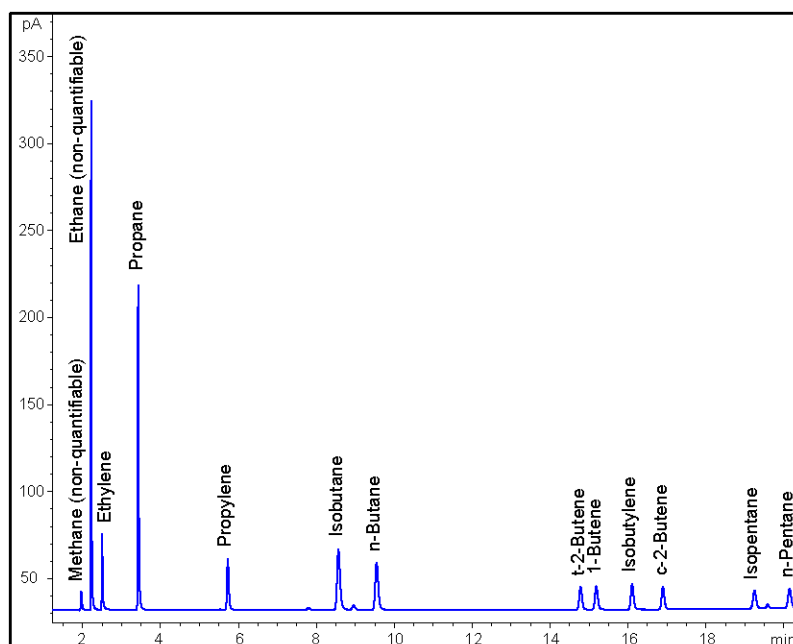


Figure 2: Hydrocarbon analysis using the Wasson-ECE Air Concentrator system and FID.

## Chromatography Tips and Tricks

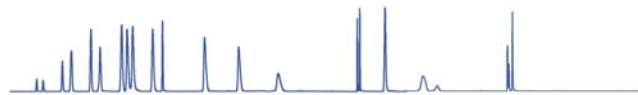
Chromatographers often ask the question “How long will my column last?” The answer to this question often lies with the user. A column’s lifespan depends on the samples, the GC system itself, and system maintenance. The following are some tips to extend the life of your column and keep your system up and running longer.

Column care and maintenance begins the moment the column box is opened. Take the following precautions to help extend the life of your column:

1. Use and regularly change gas traps to protect against the possibility of contaminated gas supply.
2. Set the GC oven to the isothermal limit of the column to ensure the upper temperature limit of the column is never exceeded.
3. When using two different columns in the same oven, make sure the isothermal limit is set to the lower of the two columns.

It is also important to consider sample composition when discussing the longevity of a column. Unwanted components in the sample include any inorganic acids or bases (even those that are neutralized) as well as any non-volatile or semi-volatile residues.

Of course, the best way to minimize system down-time and prolong column life is to ensure you have the cleanest sample possible.



When a particular sample matrix cannot be cleaned, make sure you employ the following techniques:

1. Use a liner with glass wool positioned at the bottom to keep contaminants from reaching the column.
2. Use a guard or pre-column. This is a piece of blank fused silica that collects unwanted residues before they reach the analytical column.

While there is no defined life span of a GC column, proper GC system care and maintenance can help prolong column life. Handle the column gently, take an extra moment to clean up a sample, and double check instrument parameters before starting an analysis.



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

## Wasson-ECE Instrumentation News

### 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

**Cost:** \$1000 per participant

Sign-up at [www.wasson-ece.com](http://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.

- April 14-15:** Basic GC 2-Day Course in Houston TX
- April 21:** Free Webinar on Low Level Impurities in Permanent Gases
- 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](mailto:training@wasson-ece.com) or call (970)221-9179.**



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