Quarterly Newsletter From Wasson-ECE Instrumentation:

Chromatography Corner

ISSUE 29 September 2013

this issue

upcoming events

- **Oct 15-16:** Gulf Coast Conference
  Where: Booth 1020, 1022, Galveston, TX
- **Oct 28-30:** Shanghai 8th International Petroleum Petrochemcial Natural Gas Technology Equipment Exhibition
  Where: Hall N1, Booth 601, Shanghai, China
- **March 2014:** Pittcon
  Where: Chicago, IL

For more information visit:
www.wasson-ece.com
or call (970)221-9179

Analysis of Impurities in Helium Including Hydrogen and Deuterium Quantification

Helium is the most stable element, it is inert, it cannot become radioactive, it has the lowest melting point, and it has the lowest boiling point of the elements. At temperatures close to absolute zero, helium remains a liquid. These traits make helium unique and useful in a variety of applications. Helium is used in analytical instrumentaiton, particle colliders, magnetic resonance imaging, cooled thermographic cameras, and helium-neon lasers. Helium is also a key part of many processes such as silicon and germanium crystal production, titanium and zirconium production, cooling nuclear reactors, pressurizing liquid rocket fuel, and in isotopic dating.

Because many of the trace impurities in helium do not exhibit the same unique characteristics and can negatively interfere with the application, identification and quantification of impurities in ultra-high purity helium is important. Wasson-ECE Instrumentation has configured a 7890 Agilent Technologies gas chromatograph for

the analysis of hydrogen, deuterium, krypton, xenon, oxygen, argon, nitrogen, methane, and carbon monoxide in ultra high purity helium.

Separation of hydrogen and deuterium was challenging because of their similar chemical composition and size. Separation was achieved using cryogenic cooling. Additional hardware was relocated to an auxiliary thermal zone to avoid damage to the mechanical components. The level of cryogenic cooling used to achieve separation of hydrogen and deuterium also resulted in nitrogen and argon artifacts that interfered with the analysis of impurities in helium. To overcome this interference, two methods were created.

The first method quantified hydrogen and deuterium down to a lower detection level of 1 ppm using a pulsed discharge helium ionization detector (PDHID). The second method quantified a hydrogen/deuterium composite, krypton, xenon, argon, nitrogen, methane, and carbon monoxide to a lower detection limit of 1 ppm and oxygen to a lower detection limit of 0.1 ppm using a pulsed discharge helium ionization detector (PDHID).

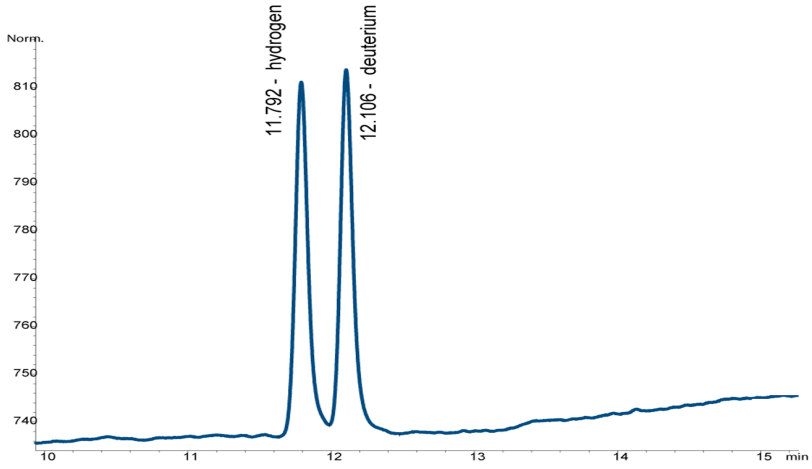

Figure 1. Hydrogen and deuterium in helium by PDHID

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Engineered Solutions, Guaranteed Results.

Auto-samplers: Reduced Labor Costs, Increased Efficiency, Greater Precision, and Safety Benefits

Automated sampling reduces the amount of manpower needed to prepare and load samples. Labor costs are one of the biggest contributors to a company's overhead. The use of auto-samplers can significantly reduce this cost. Auto-samplers can be programmed to run without employee supervision and to run after hours. Auto-samplers increase the sample volume that your test facility can handle. Many auto-samplers are even automated to perform sample preparation.

Efficiency is also increased through reduced time between injections. Auto-samplers prepare and sample faster than manual sampling. There is no lag time between runs and the next sampling event when using an auto-sampler. Samples are injected according to a set time schedule.

Auto-samplers provide more repeatable and predictable sampling. Automated sampling systems eliminate the human introduced error and human inconsistencies, resulting in increased correlation rates, tighter precision values, greater levels of sampling control, and higher accuracy rates. They are designed to maintain optimal conditions for the sample type. Sample pressures and temperatures are monitored and continually corrected. Auto-samplers aid in preventing sample phase changes, chemical decomposition, and uneven analyte distribution. Because it is a closed system, auto-samplers minimize exposure to the atmosphere and reduce sample decay.

By using an auto-sampler, the amount of employee sample handling time is decreased. The risk of dangerous accidental sample venting during sample injection is also reduced. The auto-sampler is designed to be plumbed to a safe, well ventilated outlet. The induction and expulsion of samples is performed in a closed system when using an auto-sampler. When a sample consists of highly toxic compounds, the use of an auto-sampler can improve working conditions and help protect employee health.

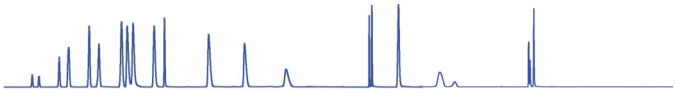
Wasson-ECE Instrumentation offers the LS100 for pressurized liquid sampling, the AS201 for gas cylinder sampling, and the Tedlar Bag Auto-sampler for gas Tedlar Bag sampling. Alternatively, Wasson-ECE maintains an engineering department ready to design custom auto-samplers for a test facilities unique needs. For additional information on custom engineered auto-samplers or a Wasson-ECE standard auto-sampler, contact the marketing department at marketing@wasson-ece.com or call (970) 221-9179.

Chromatography Tips and Tricks

The analytical column causes the analyte separations making column selection the most important decision when building a GC analyzer. Columns are selected based on compounds of interest, matrices, desired run time, injection volume, standard method compliance, and chromatogram quality. The stationary phase, column length, internal diameter, and film thickness can all affect the chromatography.

A column's inner diameter can affect the resolution, sample capacity, and speed. A smaller inner diameter will provide higher resolution and can result in a faster analysis time. The shortcoming of using a column with a small inner diameter is that it is easy to overload the column. Higher split flows must be used resulting in lower detection limit increases. Although speed will be affected by changes in inner diameter, it is not a drastic difference.

Column length can affect the speed and separations. A longer column will result in a longer analysis time. A theoretical plate is a measure of a column's separation ability. As the length of the column increases the theoretical plate count also increases because there is more stationary phase for the sample to interact with. Column length and separation ability are not directly proportional. Doubling the column length will only result in about a 40% resolution increase. If only a slight increase in resolution is required, adjusting the inner diameter or the film thickness would provide the resolution without affecting the analysis run time.



The film thickness is a measure of thickness of the stationary phase covering the wall of the capillary column. A thicker film lowers the maximum operating temperature, widens the peaks, and results in more column bleed. The benefits of a thicker film include higher sampling capacity, increased separation, and reduced unwanted sample interactions with the column tubing. A thinner film will result in increased peak sharpness and reduced column bleed which will both contribute to lower detection limits. A reduced film thickness will also allow for faster analyte elution at lower temperatures.

The stationary phase is the portion of the analytical column that interacts with the sample to create the separations. The chemical properties of each stationary phase interact differently with the same samples and analytes causing different separations for different columns. The polarity of a stationary phase is the strongest influence in determining analytical separation. Non-polar columns work through dispersion and separate components by size and boiling point. Polar columns separate components through dipole, p-p, and acid-base interactions.

By selecting a different stationary phase, adjusting the column length, selecting an alternative film thickness, and choosing a different size inner diameter, chromatography can be altered to maximize component separations, resolution, lower detection limits, and analysis run times.

For more information on column selection, contact the Wasson-ECE technical service department at (970) 221-9179.

Wasson-ECE Instrumentation News

Wasson-ECE Expands Virtual Application Notes

Wasson-ECE has recently updated our website to include new application notes that highlight analyzer descriptions, chromatography examples, key features and benefits, and additional literature references.

Some of the most recent application notes include:

- ISO 6974-6:2002
- Analysis of Volcanic Gas
- ASTM D6229
- ASTM D3257

Is there an application you would like to learn more about or see on the website? Email sales@wasson-ece.com 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 30 years of experience can make.

Events Calendar

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