

Chromatography Corner

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upcoming events

- **May 15-17:** CISLE, Booth 1108
Where: Beijing, China
- **June 23-27:** ASTM
Where: Beijing, China
- **Oct 15-16:** Gulf Coast Conference
Where: Galveston, TX

For more information visit:
www.wasson-ece.com
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Analysis of Amines in Ethylene, Propylene, Butane and Pentane Streams

Amines are frequently used as reactive intermediates in the synthesis of various organic chemicals during hydrocarbon processing. Wasson-ECE Instrumentation configured an Agilent Technologies gas chromatograph with a nitrogen chemiluminescence detector (NCD) for the analysis of residual trace amines in propylene, ethylene, butane, and propane.

To accommodate the different matrices, the gas chromatograph was configured with both a liquid sample valve and a gas sample valve. An automatic liquid sampler could also be used. Components analyzed on the NCD include methylamine, dimethylamine, trimethylamine, ethylamine, diethylamine, propylamine, pyridine, aniline, quinolone, and indole to a lower detection limit of 0.5 ppm.

Separation between dimethylamine, trimethylamine, and ethylamine was difficult to achieve. In order to maximize resolution of all components and meet the lower detection limits, two methods were developed. The first method detects a composite of light amines, aniline, quinolone, and indole in approximately fifteen minutes. The second method analyzes methylamine, dimethylamine, trimethylamine, ethylamine, diethylamine, propylamine, and pyridine in approximately fourteen minutes. In the second method the heavier nitrogen-containing components are backflushed to vent to prevent column degradation.

The instrument was configured with specially passivated hardware to improve peak shape and reduce tailing. By customizing the hardware for trace nitrogen-containing components and developing two methods, Wasson-ECE Instrumentation was able to quantify the full spectrum of amines in less than half an hour.

Figure 1. Heavy amine analysis

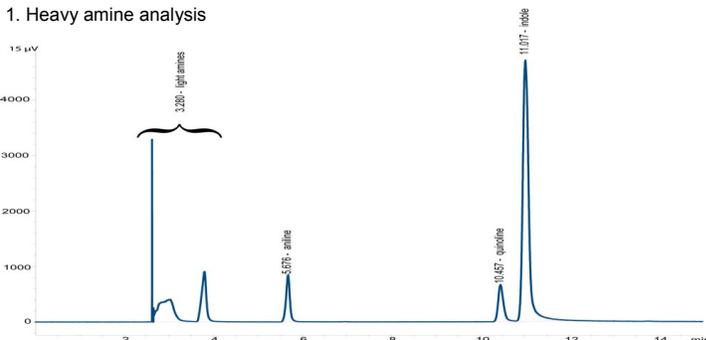
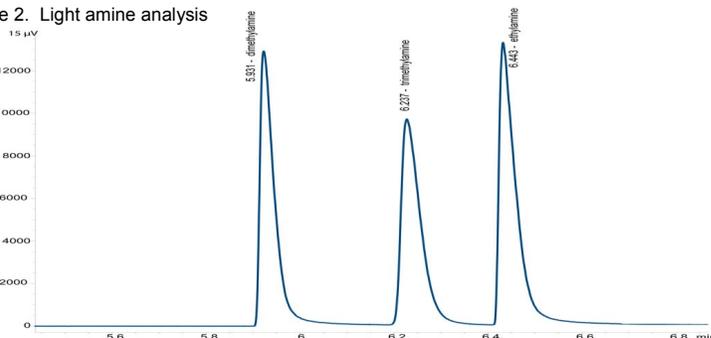


Figure 2. Light amine analysis



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Analysis of Impurities in Various High Purity Gases

High purity gases are critical to the operation of many different industries including scientific analytics, medicine, pharmaceuticals, and manufacturing for photovoltaics, flat panel displays, semiconductors, and medical devices. The use of high purity gases can improve yield, optimize performance, and lower costs. Wasson-ECE Instrumentation has customized an Agilent Technologies gas chromatograph to identify and measure impurities in carbon dioxide, helium, hydrogen, argon, oxygen, carbon monoxide, nitrogen, and methane.

Wasson-ECE Instrumentation was able to combine the analysis of eight different matrices into one instrument by creating seven different methods. This analyzer will save money and space with its versatility and user-friendly application design. Switching matrices is as simple as loading a new method, purging the sample lines with helium, and running a few blank injections.

The gas chromatograph was configured with dual pulse discharge helium ionization detectors (PDHID/PDHID). The instrument was built with onboard high purity regulators, helium purifier, specially passivated tubing, gold ferrules, and diaphragm valves to minimize atmospheric exposure and ensure the lowest detection limits possible.

The first PDHID detected hydrogen, argon, oxygen, nitrogen, methane, and carbon monoxide to a lower detection limit of 50 ppb. It detected krypton to a lower detection limit of 200 ppb. The second PDHID used a second injection and a different set of columns to heart cut carbon dioxide away from the matrix to provide additional separation. This dual detector configuration allows Wasson-ECE to achieve high quality resolution and a lower detection limit of 50 ppb for carbon dioxide. Competitor's single detector configurations can only reach 1 ppm lower detection limit and carbon dioxide elutes on the tail of the matrix. Analysis of impurities in high purity gas is a challenge because components of interest can elute on the tail of the matrix or co-elute with the larger matrix peak. Wasson-ECE Instrumentation overcame this challenge by using dynamic chromatography to perform heart-cuts where a small aliquot containing a specific analyte and a small amount of matrix gas is transferred to a second set of analytical columns. The separation is achieved when the quantity of matrix that the analyte must be separated from is drastically reduced.

Wasson-ECE Instrumentation was able to use their 25 years of experience in multi-dimensional chromatography to accurately separate impurities in eight different high purity gas matrices on one gas chromatograph to a lower detection limit of 50 ppb in less than twenty minutes per matrix.

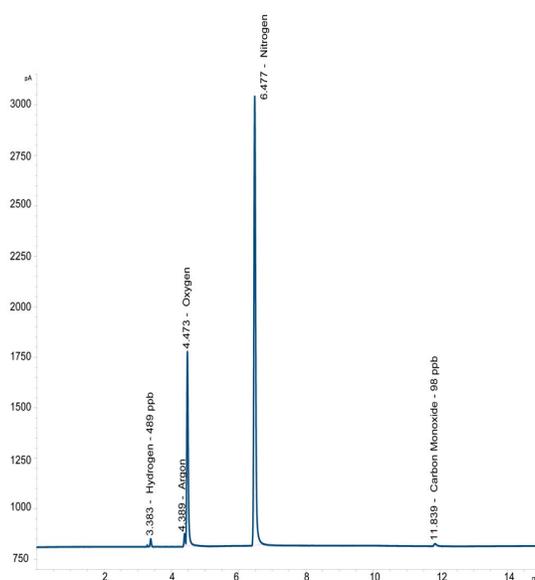


Figure 3. Permanent gas analysis

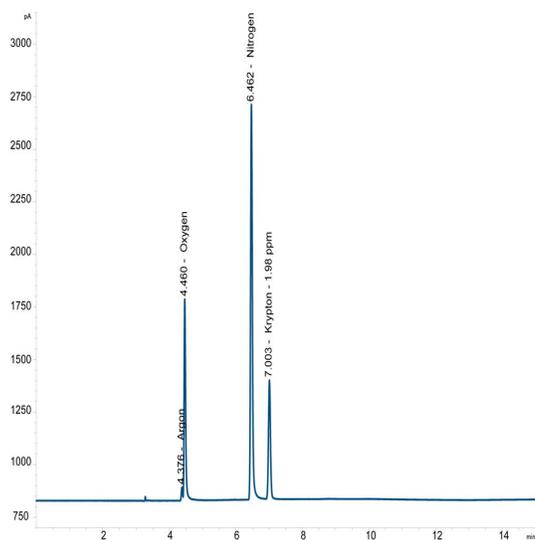


Figure 4. Noble gas analysis

Chromatography Tips and Tricks

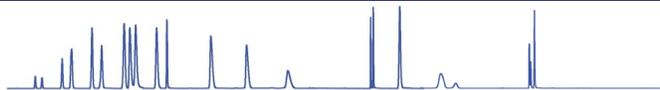
In honor of National Reading Month, Wasson-ECE Instrumentation would like to share a few of our favorite books.

Basic Gas Chromatography by Harold McNair and James Miller (New York: John Wiley & Sons, Inc., 1998)

Basic Gas Chromatography was written by two internationally known chromatographers with extensive practical experience in the field. This book is a useful introduction to the applied principles of gas chromatography, as well as a handy reference for the more experienced chromatographer. It is practical, easy to follow, and avoids bogging the reader down with theoretical equations and derivations. The book touches on basic trouble-shooting, GC-MS, headspace analysis, chiral analysis, and solid phase microextractions. However, for the advanced chromatographer other literature will be more useful in developing complex analytical techniques.

Modern Practices in Gas Chromatography, 4th ed. edited by Robert Grob and Eugene Barry (Hoboken: John Wiley & Sons, Inc., 2004)

Some people have referred to *Modern Practices in Gas Chromatography* as the bible of gas chromatography. Wasson-ECE recommends this book as a follow-up to *Basic Gas Chromatography*. *Modern Practices in Gas Chromatography* is a more comprehensive look into the science with more theoretical depth, detailed analysis of available hardware, and thorough introductions to modern techniques in chromatography. The analytical column is at the heart of gas chromatography and this book acknowledges that with extensive explanations of the types and functions of both capillary and packed columns. For the more experienced chromatographer, this book offers sections on gas chromatography/mass spectrometry (GC/MS); optimization of separations and computer



assistance; high speed or fast gas chromatography; mobile phase requirements; gas system requirements and sample preparation techniques; qualitative and quantitative analysis by GC; and, validation and QA/QC of chromatographic methods.

The Troubleshooting and Maintenance Guide for Gas Chromatographers by Dean Rood (Weinheim: WILEY-VCH Verlag GmbH & Co. KGaA, 2007)

The information contained in *The Troubleshooting and Maintenance Guide for Gas Chromatographers* encompasses nearly 25 years of the author's in-depth field experience along with the wisdom collected from GC practitioners around the world. Practical information mixed with a touch of theory often proves to be the most useful and helpful when problems are encountered in the laboratory. This book is chock full of pertinent information and explains gas chromatography in real-world practical terms. The book is structured in a manner that makes information easy to locate in a trouble-shooting emergency. It is the most concise and applied of the books recommended here and a must have for every laboratory shelf.

Analytical Gas Chromatography, 2nd ed. by Walter Jennings, Eric Mittlefehldt, and Philip Stremple (San Diego: Academic Press, 1997)

Analytical Gas Chromatography provides a detailed look at the history and theory of gas chromatography. It details an in-depth approach to the theoretical and scientific principles with the complex equations and derivations available to explain the application of principle to the science gas chromatography. To demystify the "black box" magic of gas chromatography and gain a full understanding of the science that governs the application of gas chromatography, this book is an excellent choice.



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:

- Analysis of Nitrogen Containing Compounds in Ethylene, Propylene, Butane, and Pentane Streams
- Analysis of Impurities in Helium including Hydrogen and Deuterium Quantification

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.



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.

May 15th-17th: China International Scientific Instrument and Laboratory Equipment Exhibition in Beijing, China, Booth 1108

June 23rd-27th: ASTM Annual Meeting in Montreal, Canada

October 15th-16th: Gulf Coast Conference, Galveston, TX

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