Monthly Newsletter From Wasson-ECE Instrumentation:

Chromatography Corner

ISSUE 12 December 2009

this issue

upcoming events

- **January 20:** Free El Paso Stripper 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

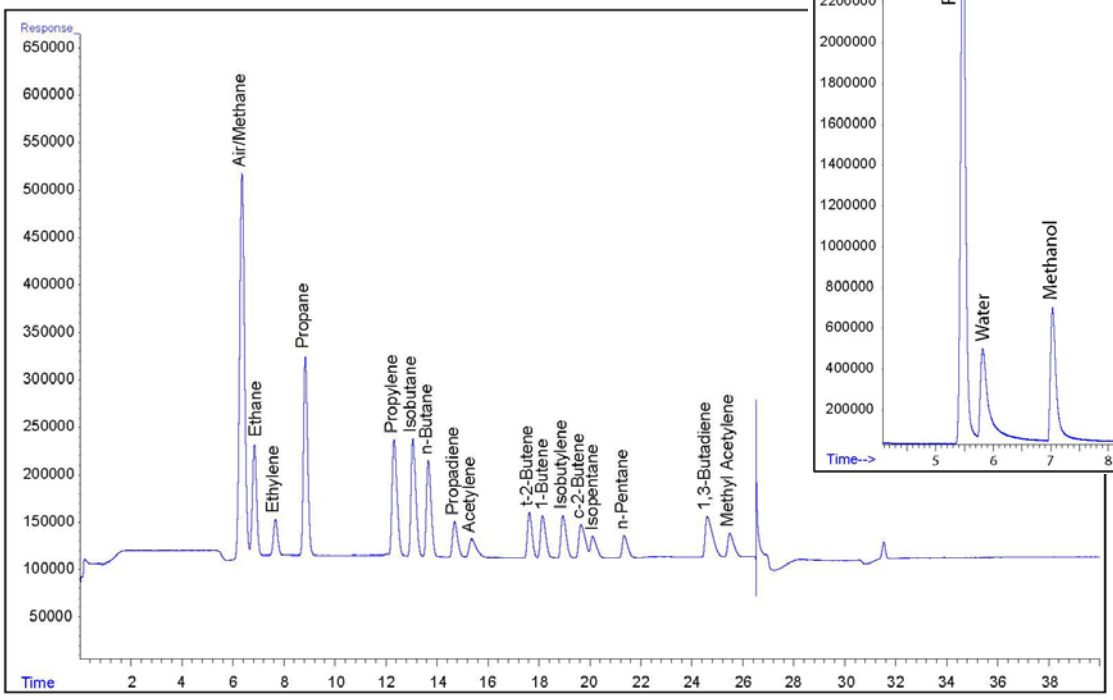
Custom TCD/TCD/MSD Analysis of Refinery Gas and Liquid Mixtures

For the analysis of refinery gas and liquid mixtures, Wasson-ECE customized an Agilent Technologies GC with dual thermal conductivity detectors (TCD/TCD) and a mass selective detector (MSD).

Components analyzed on TCD 1 included carbon dioxide, ethylene, ethane, acetylene, hydrogen sulfide, an argon/oxygen composite, nitrogen, methane, and carbon monoxide to 200 parts per million (ppm) except for carbon monoxide which had a lower detection limit (LDL) of 400 ppm. TCD 2 detected hydrogen to a LDL of 100 ppm.

Components identified on the MSD included formaldehyde, methanol, and water. The CAMS (Constant Aspiration Mass Spectrometry) analysis used a vacuum to aspirate a stream of gas. This analysis was used to monitor the customer's reactor in real-time while the GC/MS was not being utilized

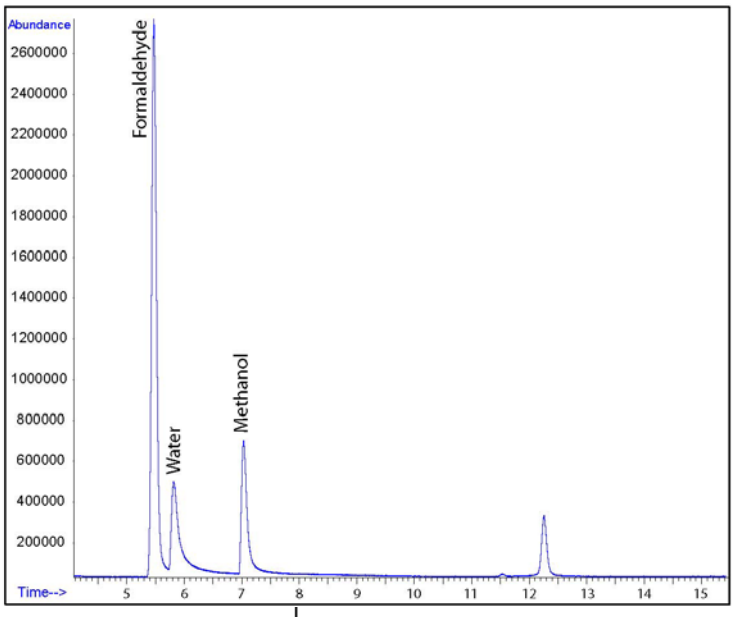
Figure 1: Hydrocarbon analysis by MSD using Wasson-ECE refinery gas standard at percent levels.



The GC was configured to introduce samples to the separation columns by gas sample valve or syringe injection of ambient liquid at the front inlet.

The most challenging part of the analysis was quantifying formaldehyde, methanol, and water by MSD. For this analysis the components were separated by polarity. Although Total Ion Chromatogram (TIC) exhibits some chromatographic overlap, these components are readily separated by extracting unique target ions (m/z): formaldehyde 29, methanol 31, and water 18. The analysis was completed in approximately 20 minutes.

Figure 2: Analysis of formaldehyde, water and methanol by MSD.



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Dual GC Systems for the Simultaneous Analysis Of Fuel Cell Reactor Effluent Gas

Kerosene or diesel oil are placed into a hydrogen generator to produce fuel cell hydrogen. Since hydrogen quality is critical, the hydrogen generator process must be closely monitored.

A combination of two gas chromatograph (GC) systems were customized by Wasson-ECE for the analysis of fuel cell reactor effluent gas. The Agilent Technologies GCs were tied to a single gas sample injection system, which injected four simultaneous aliquots. Chromatograph A was a TCD/TCD/FID system for the analysis of "refinery gas" type components to a lower detection limit (LDL) ranging from 10-400 ppm. Components analyzed on the FID included paraffins and olefins. Components identified during method development included: methane, ethane, ethylene, propane, propylene, acetylene, isobutane, propadiene, n-butane, trans-2-butene, 1-butene, isobutylene, cis-2-butene, neopentane, isopentane, methyl acetylene, n-pentane, 1,3-butadiene, neo-hexane, n-hexane, n-heptane and benzene with an initial C_8^+ backflush to the detector.

Components analyzed on TCD 1 included carbon dioxide, ethylene, ethane, acetylene, hydrogen sulfide (H_2S), an argon/oxygen composite, nitrogen, methane, and carbon monoxide. TCD 2 detected hydrogen.

Chromatograph B was an FID/FPD system for the analysis of C_5 to approximately C_{20} including n-pentane, n-hexane, n-heptane, n-nonane, n-decane, n-undecane, n-dodecane, n-hexadecane, n-octadecane, and eicosane by FID in a simulated distillation fashion and H_2S by FPD to a LDL of 50 ppm.

The most difficult part of this analysis was that the samples had to be kept at 350°C. Traps were placed inline to remove heavy waxes from the streams to ensure lines and electronic pressure control (EPC) modules did not get clogged. These traps were not placed on the heavy hydrocarbon FID analysis.

By customizing two GC systems, Wasson-ECE was able to perform a comprehensive analysis of paraffins, olefins and H2S in fuel cell reactor effluent gas.

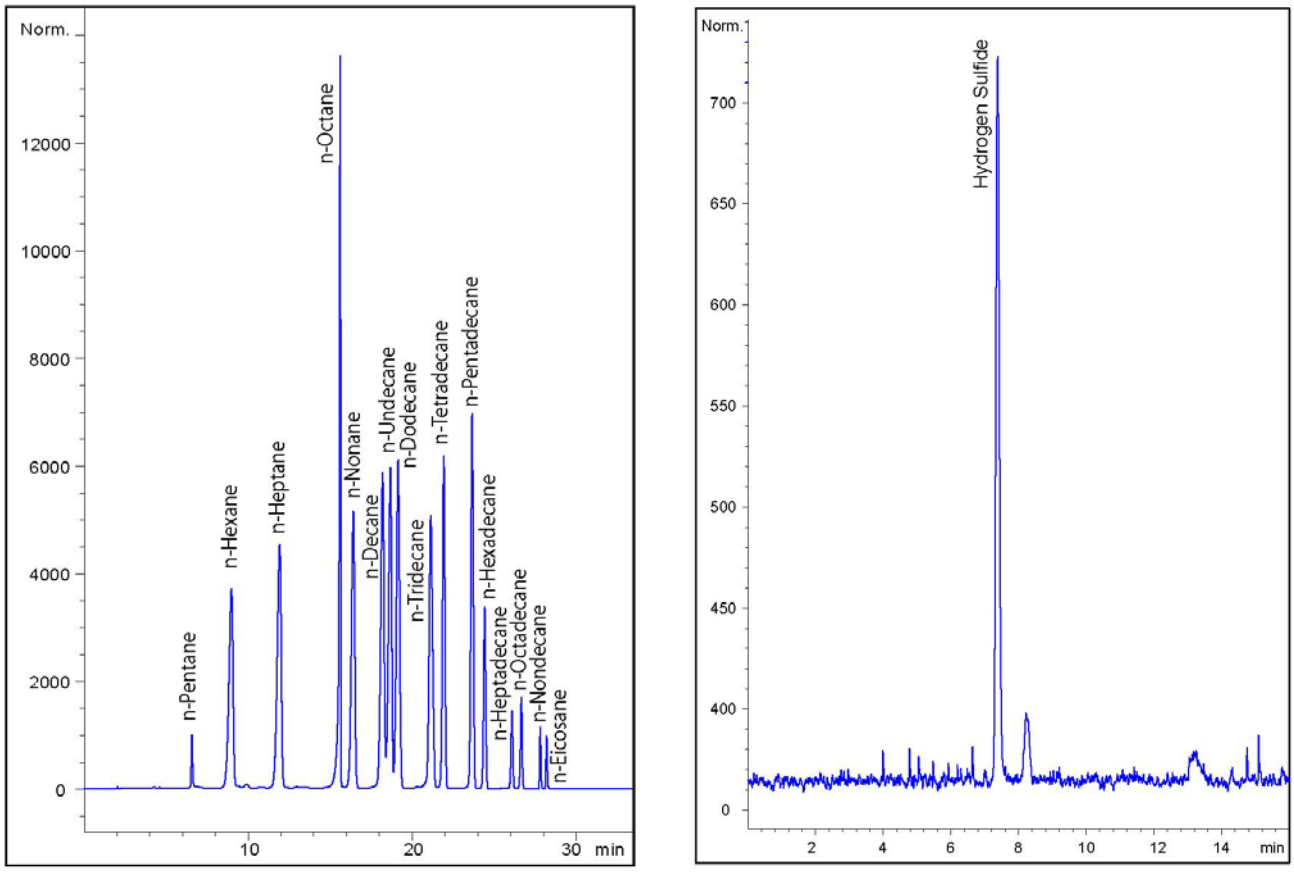

Figure 3 and 4: C_5-C_{20} n-paraffin analysis (% levels) by FID and hydrogen sulfide analysis at 50 ppm by FPD.

Chromatography Tips and Tricks

As 2009 comes to an end, Wasson-ECE would like to share some of our best gas chromatography tips of 2009.

1. If the inlet temperature is not specified in a method, 250°C is a good staring point.
2. To prevent retention times from shifting, replace your septum every 50 to 100 injections.
3. Make sure to change gas cylinders when tank pressure reaches 200 psi to avoid non-volatiles, such as hydrocarbons, from reaching GC flow paths.
4. Always use the appropriate carrier gas traps and purifiers for best results.
5. Purge the GC column with a carrier gas, free of oxygen and moisture for 15-20 minutes before heating the column. This removes any air that may be detrimental to the analysis.
6. Select the least polar column with adequate separation for your analysis. The more polar the columns is, the more susceptible it is to degradation.
7. To extend the life of a polar column, be sure to use an oxygen trap inline with the carrier gas.
8. Always cut the ends of a capillary column after inserting through graphite ferrules
9. If early eluting peaks are broad or distorted, consider using a column with a thicker film.
10. Always check the maximum temperature of all components and columns in the oven before conditioning. It may be necessary to do some "baking out" processes in another oven.
11. To reduce peak fronting try injecting less volume, diluting the sample with a solvent, or increasing the split ratio.
12. Use a pre- or stripper column when samples contain heavy non-volatile residues that may contaminate the analytical column.
13. Do not thermally shock a column by disconnecting it while hot or under carrier gas pressure. Allow to cool to an ambient temperature and slowly bleed the pressure off.

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

Question of the Month

Your sample stream has a matrix of argon and requires that you analyze sulfur compounds to 75 ppb. What would be your best detector choice, keeping in mind that you want to keep costs low and do the minimal amount to maintenance?

A. Sulfur Chemiluminescence Detector (SCD)

B. Pulsed Flame Photometric Detector (PFPD)

C. Flame Photometric Detector (FPD)

Enter for a chance to win a digital camera for your lab. One winner will be chosen quarterly from a random drawing from the correct answers received. Answers to the monthly question can be faxed to 970-221-9364, emailed to QOM@wasson-ece.com or mailed to 101 Rome Court, Fort Collins, CO, 80524, Attention: Marketing.

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 TCEQ modified El Paso Method
February 17: Free Dynamic Blender 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 Tedlar Bag Autosampler Webinar
May 26: Free Webinar on New Wasson-ECE Hardware TBD
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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