Monthly Newsletter From Wasson-ECE Instrumentation:

Chromatography Corner

ISSUE 15 March 2010

this issue

upcoming events

- **March 31:** Free Automator Webinar
  Time: 9:00 am MST
- **April 14-15:** Basic GC 2-Day Course
  Where: Houston, TX
  Cost: $1000 per person

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

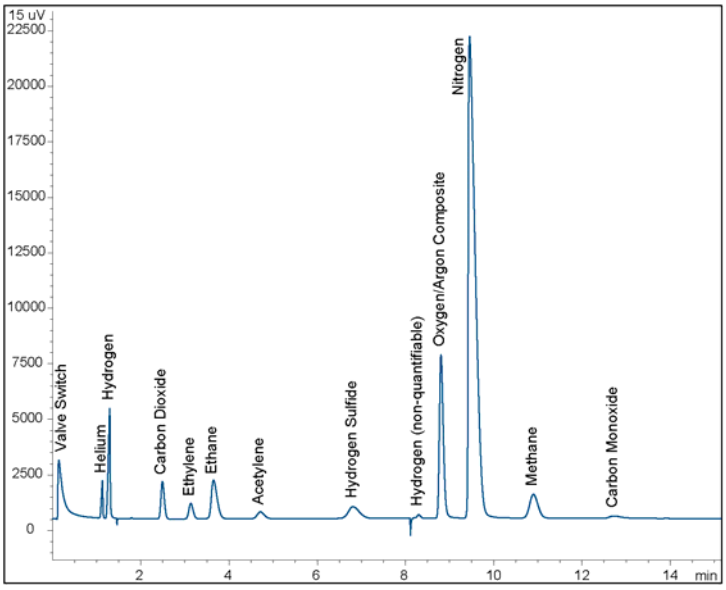
Analysis of Acid Gas by TCD/TCD/FID

Acid gas refers to natural gas or gas mixture containing significant amounts of hydrogen sulfide (H_2S), carbon dioxide (CO_2) and other contaminants. Before raw natural gas containing H_2S, CO_2 and other impurities can be used, the gas must be treated to reduce contaminants to acceptable levels. This treatment is commonly done with an amine gas treating process, which is referred to as gas sweetening. Not only is H_2S toxic, but it also limits the type of materials that can be used for piping and other equipment used for handling the natural gas, as many metals are sensitive to sulfide stress cracking.

For the analysis of acid gas, Wasson-ECE configured an Agilent Technologies GC with dual thermal conductivity detectors and a flame ionization detector (TCD/TCD/FID).

Components quantified by FID included C1-C5 paraffins and olefins to an LDL of 20 ppm except for trace peaks eluting on the tail of a major component. TCD 1 quantified CO_2, oxygen/argon composite, nitrogen, and methane to an LDL of 200 ppm, carbon monoxide to an LDL of 400 ppm and H_2S to an LDL of 500 ppm. TCD 2 detected hydrogen to a LDL of 100 ppm.

Due to the need for an extremely fast analysis time of 6 minutes, partial co-elutions of C_5 olefins occurred on the FID train.



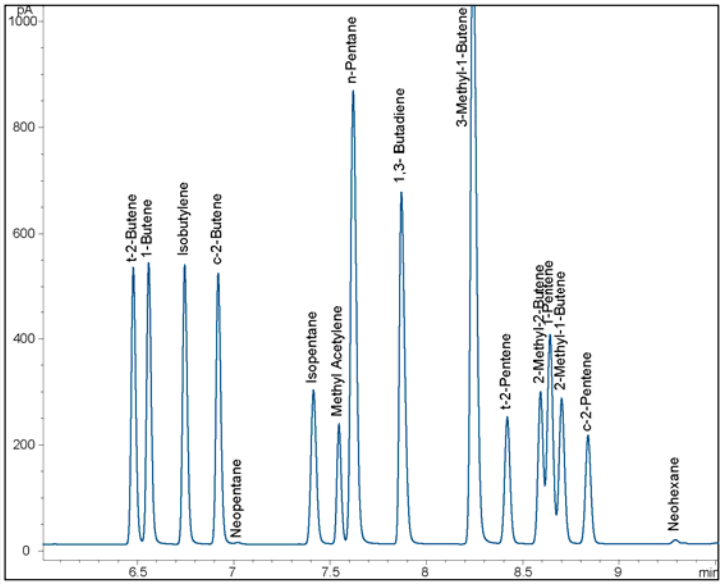


Figure 1: Permanent gas analysis by TCD
Figure 2: Hydrocarbon analysis showing an enlarged area of C5 impurities by FID

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Analysis of Impurities in Chlorine

Chlorine is used in a variety of industrial and consumer products such as plastics, solvents for dry cleaning, metal degreasing, textiles, agrochemicals, pharmaceuticals, insecticides, and household cleaning products. Due to the corrosive nature and potential health effects of chlorine, special considerations and precautions must be taken to ensure the safety and longevity of the gas chromatograph (GC) system.

Wasson-ECE configured an Agilent Technologies GC with dual thermal conductivity detectors (TCD/TCD) for the analysis of impurities in chlorine. Samples were analyzed in the gas phase. TCD 1 detected carbon dioxide, oxygen/ argon composite, nitrogen, methane, carbon monoxide to a lower detection limit (LDL) of 200 parts per million (ppm) except for carbon monoxide, which was 400 ppm. TCD2 detected hydrogen to a LDL of 200 ppm.

Since chlorine is extremely corrosive, and can ruin GC components quickly, Wasson-ECE used Hastelloy valves and nickel tubing.

The primary function of Hastelloy is that it is effective under high temperature and highly corrosive environments. The predominant alloying ingredient is typically nickel. Other alloying materials are added to the nickel including molybdenum, chromium, cobalt, iron, copper, manganese, titanium, zirconium, aluminum, carbon, and tungsten.

To ensure the detector was not damaged during the analysis of chlorine, a proprietary stripper column was installed before the analytical column. This column trapped the chlorine and allowed the impurities of interest to elute to the analytical column and detector.

By taking special precautions such as Hastelloy valves, nickel tubing, and the use of a proprietary stripper column, Wasson-ECE was able to detect and quantify impurities in chlorine to ppm levels.

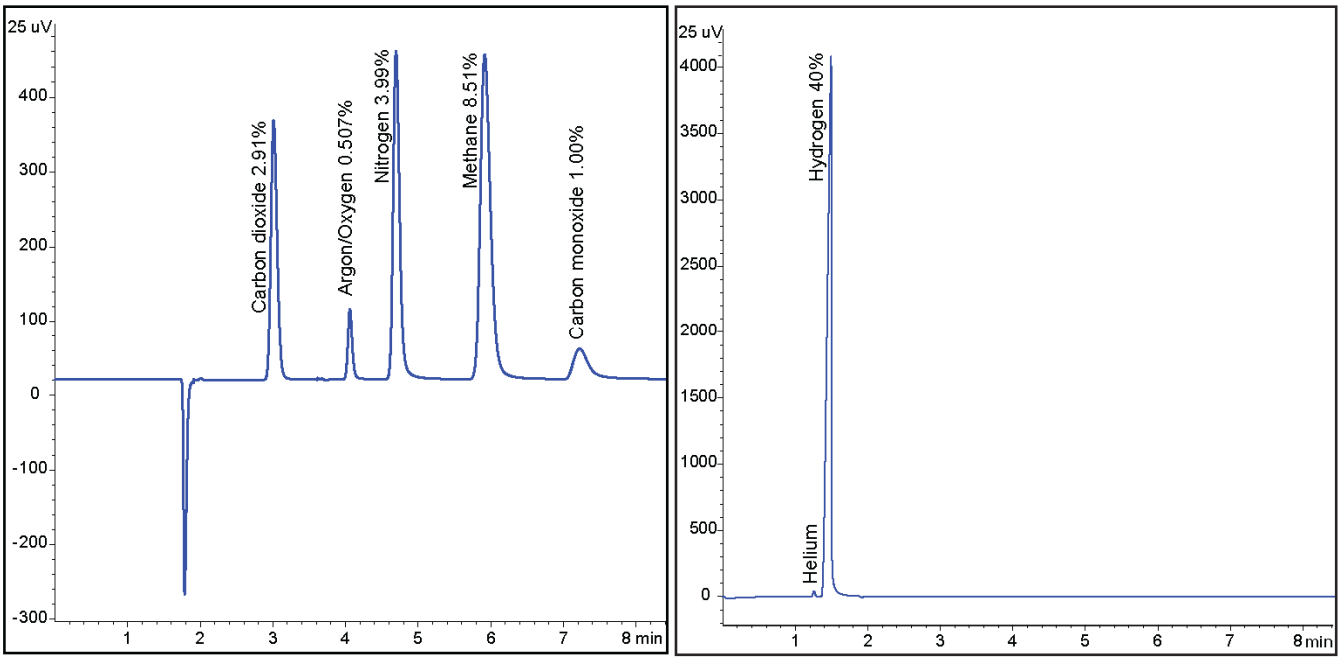
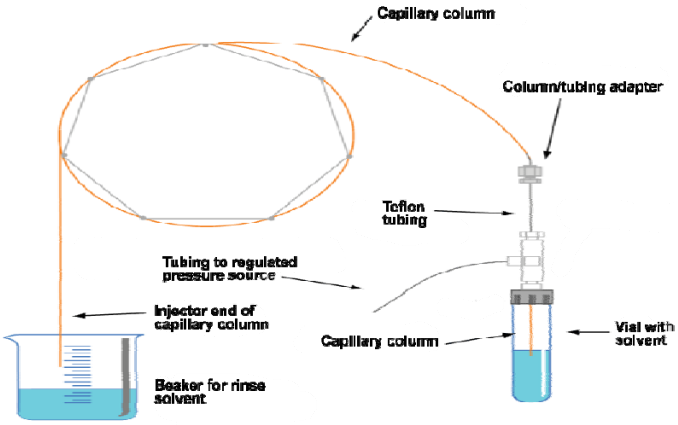


Figure 3 and 4: Analysis of impurities in chlorine by TCD at percent levels.

Chromatography Tips and Tricks

When column contamination can not be removed by baking out or trimming the column it may be necessary to solvent rinse the column. Any stubborn residues that are soluble in the solvent are washed from the column. Injecting large volumes of solvent while the column is still installed is not rinsing a column nor does it remove contaminants. **Note: A capillary GC column must have a bonded and cross-linked stationary phase before it can be solvent rinsed. Rinsing a non-bonded stationary phase results in severe damage to the column.**

Multiple solvents are normally used to rinse a column. Each successive solvent must be miscible with the previous one. High boiling point solvents should be avoided especially as the last solvent rinse. A good choice for the first rinse is the sample matrix. A series of solvents that works well in the majority of cases is methanol, followed by methylene chloride, and ending with hexane. Acetone can be substituted for methylene chloride to avoid using a halogenated solvent.



If samples are aqueous based, such as biological fluids and tissues use water prior to the first methanol rinse.

The table below suggests solvent volumes for different diameter columns. Using larger solvent volumes is not harmful, but rarely beneficial. After adding the first solvent, pressurize the solvent through the column, but stay below 20 psi. Use the highest pressure that keeps the solvent flow rate below 1 mL/min. Longer rinse times are required when using heavy or viscous solvents, and for longer or smaller diameter columns. When most of the first solvent has entered the column, add the next solvent. The previous solvent does not have to vacate the column completely before the next solvent is started. After the last solvent has left the column, allow the pressurizing gas to flow through for 5-10 minutes. Install the column into the injector, and turn on the carrier gas. Allow the carrier gas to flow through the column for an additional 5-10 minutes. Attach the column to the detector and using a temperature program starting at 40-50°C, heat the column at 2-3°/min until the upper temperature limit of the column is reached. Maintain the upper temperature limit for 1-4 hours until the column is fully conditioned.

Column I.D. (mm)	Solvent Volume (mL)
0.18-0.2	3-4
0.25	4-5
0.32	6-7
0.45	7-8
0.53	10-12
*Using larger volumes will not damage the column.	

Additional questions? Contact our service department at (970)221-9179 or 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 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

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 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 or call (970)221-9179.

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