Monthly Newsletter From Wasson-ECE Instrumentation:

Chromatography Corner

ISSUE 02 FEBRUARY 2009

this issue

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upcoming events

- **Feb 25:** PNA Webinar
  Time: 9:00am MT
- **March 25:** Variable Pressure Sample System Webinar
  Time: 9:00am MT

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

Analysis of Beverage Grade Carbon Dioxide

Carbon dioxide, used in the production of carbonated soft drinks and other beverages, can be produced from a variety of sources including ammonia, fermentation, chemical plants, and oil refineries. This variety of production sources results in an array of possible chemical contaminants which can be found in the carbon dioxide. In addition, companies are now limited by industry stipulations on the maximum levels of these impurities.

The impurities that must be assessed and controlled include hydrogen, oxygen, nitrogen, oxides of nitrogen, oxygenated hydrocarbons, sulfur compounds, phosphine, cyanide, chlorinated compounds, ammonia, light paraffins, olefins, non-volatile hydrocarbons, benzene and other aromatic hydrocarbons. Because of the wide variety of compounds that must be analyzed, a gas chromatograph (GC) is an excellent tool for the analysis, with its ability to separate and quantify a range of compounds.

Wasson-ECE Instrumentation customized two GCs to quantify trace level impurities in beverage grade carbon dioxide. The first system was configured with dual flame ionization detectors (FID) and a mass selective detector (MSD). The FIDs quantified volatile paraffins and olefins, as well as heavy hydrocarbons. The MSD quantified benzene, toluene, ethylbenzene and xylenes (BTEX).

Using the Selected Ion Monitoring (SIM) mode, the MSD provides conclusive component concentration measurement in the parts-per-billion (ppb) range. SIM allows the MSD to be programmed to respond only to a known molecular fragmentation pattern or spectrum at any given time, so it can confirm the presence of each analyte in question rather than relying on retention time only.

The second system was configured with a sulfur chemiluminescence detector (SCD) and cryogenic cooling to quanify trace level sulfur compounds in the low part-per-million (ppm) range. A pulsed discharge helium ionization detector (PDHID) was also included on the second system to detect oxygen, argon, nitrogen, carbon monoxide, and methane in the low ppm range.

The carbon dioxide samples could be introduced to the GC as a pressurized liquid or gas. Selection valves were provided to direct the flow to the appropriate sample input port. In the case of pressurized liquid, an internal vaporizer converts the sample to gas phase for analysis.

With strict standards for carbon dioxide, beverage companies need a reliable and accurate testing method to assure purity. Using dual GCs, Wasson-ECE has created a complete analysis for trace impurities in beverage grade carbon dioxide.

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Ammonia Analysis in Refinery Gas

During catalytic cracking, ammonia (NH_3) is able to form if nitrogen containing components are present in the feedstock. A water wash is then used to remove the NH_3 from the stream leaving a pure product. Because of the inability of the water to completely remove NH_3, it must be quantified to low level parts-per-million (ppm) range.

For the quantification of NH_3 Wasson-ECE Instrumentation reconfigured an Agilent 6890N Refinery Gas Analyzer with a nitrogen chemiluminescence detector (NCD) for gas phase samples. With this highly specific detector, NH_3 was quantified to a lower detection limit (LDL) of 1 ppm. However, NH_3 is nonpolar and contains a non-bonding electron, which reacts with metal surfaces resulting in poor peak shape and tailing. To help improve peak shape, sample lines were nickel plated and a special base deactivated capillary column was used for separation. In addition, NH_3 reacts with hydrogen sulfide (H_2S) to form ammonium hydrosulfide (NH_4HS) salt.

As a result H_2S was quantified on a separate Agilent 6850 Series GC with a flame photometric detector (FPD) to a LDL of 0.5 ppm.

Due to the radically different oven temperatures needed to quantify ammonia and refinery gas impurities, two methods were created for the complete analysis. The first method was used for NH_3 analysis, while the other method quantified the impurities in refinery gas samples. The refinery gas analysis (RGA) was mostly unchanged from its original state, leaving the dual TCDs and FID to detect carbon dioxide, ethylene, ethane, acetylene, argon/oxygen composite, nitrogen, methane, carbon monoxide and hydrogen, as well as C1-C7 paraffins and olefins.

This system ensured a pure product free of trace NH_3 and other refinery gas impurities by using the traditional refinery gas analysis and adding an NCD.

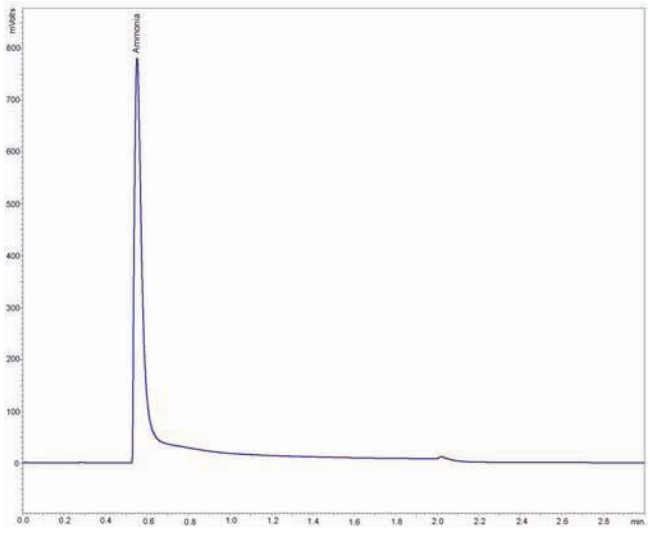

Figure 1: 5 ppm ammonia Wasson-ECE standard in nitrogen analysis using an NCD.

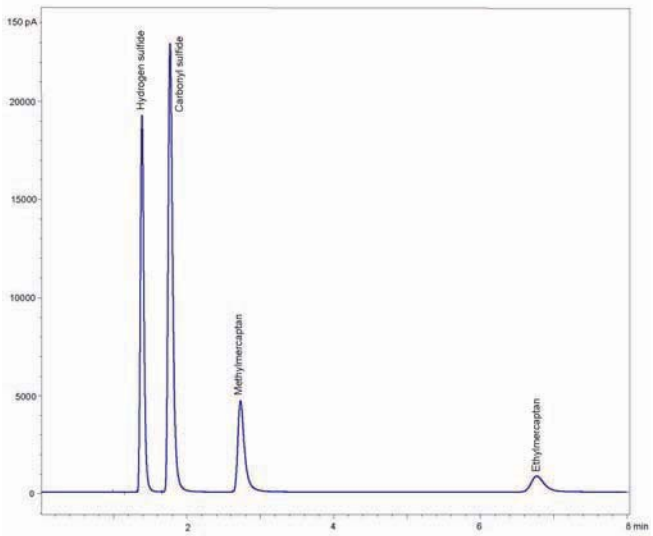

Figure 2: 10 ppm sulfur component standard on an FPD.

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Chromatography Tips and Tricks

For a chromatographer, poor peak shape is often unacceptable. Peak tailing makes quantification of other components in the sample stream difficult. There are a number of reasons for peak tailing including loss of column phase, incorrect column for the sample, and a poorly installed capillary column.

The sudden appearance of a tailing peak can be a signal that there has been a significant loss of phase on the column. A loss of phase causes more active sites on the fused silica and means there are fewer interactions between the stationary phase and the sample stream. In addition to peak tailing, shorter retention times can also be a sign there has been a loss of phase. Besides changing out the damaged column, peak tailing can be minimized by using temperatures below the maximum operating temperature of the column and not leaving the column at high temperatures for excessive amounts of time.

A loss of phase can happen over time, but peak tailing that appears from the very first injection can signify a few different problems. If the sample is incompatible with the column phase, peaks may tail. Analytes that are being quantified should be examined and an appropriate column should be chosen. For example polar components are more likely to tail if they are analyzed on a non-polar column due to fewer interactions with the stationary phase.

A better column choice would be one that is more polar with a thicker film to increase sample and phase interactions.

Another situation that can cause peak tailing is an incorrectly installed capillary column. A capillary column that is installed too low in the injector can result in dead volume and slow transfer of components to the front of the column. A correctly installed capillary column should be 4-6 mm above the ferrule on the inlet side. By eliminating dead volume, components will quickly be swept onto the capillary column and should prevent peak tailing.

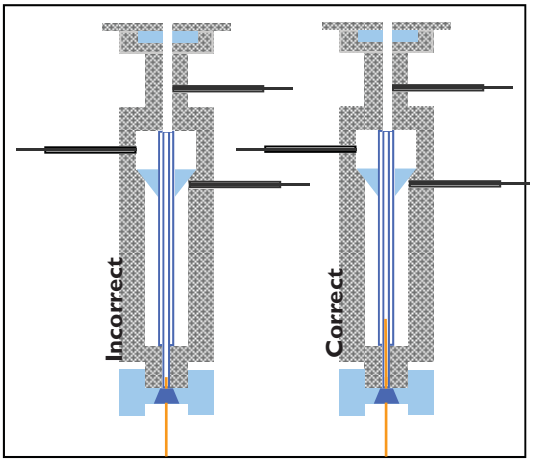

Figure 3: Diagram of incorrectly and correctly installed capillary column into a split/splitless injector.

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

Question of the Month

Q: Various carrier gases can be used with a thermal conductivity detector. In order to determine which carrier gas to use, you must know what components you want to analyze and then study the thermal conductivities of the components as compared to the carrier gas. The components of interest are hydrogen, nitrogen, oxygen and carbon monoxide. What carrier gas would be best for this analysis?

Enter for a chance to win a digital camera for your lab. One winner will be chosen randomly from the correct answers. 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.

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specializes in configuring and modifying new or existing Agilent Technologies gas chromatographs. ur 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 20 years of experience can make.

E ents alendar

February 2 Free PNA Webinar
arch 2 Free ariable Pressure Sample System Webinar
April -9 DHA Training at Wasson-ECE in Fort Collins, C
April 22-23 Basic GC Course at Wasson-ECE in Fort Collins, C
ay 20 Free Automator Webinar
une 24 Free Blender Webinar
uly 29-30 Basic GC Course at Wasson-ECE in Fort Collins, C
August 26 Free PNA Webinar
eptember 16-17 Lab anagers Training at Wasson-ECE in Fort Collins, C
eptember 23 Free xy RGA Webinar
ctober 21-22 Basic GC Course at Wasson-ECE in Fort Collins, C
ctober 2 Free Webinar TBD

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