Quarterly Newsletter From Wasson-ECE Instrumentation:

Chromatography Corner

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upcoming events

- **Oct 16-17:** Gulf Coast Conference 2012
  Where: Booth 621 & 623 Galveston, TX
- **March 17-21:** Pittcon 2013
  Where: Philadelphia

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

Analysis of Flue Gas for Pollution Control

Flue gas is formed during the combustion of fossil fuels and is exhausted into the atmosphere. Flue gas monitoring is an important part of pollution and greenhouse gas reduction strategies. Monitoring of flue gases also measures the efficiency of a combustion process. The typical makeup of flue gas is 78-80% nitrogen, 8-14% carbon dioxide, 2-6% oxygen, 80-150 ppm carbon monoxide, 50-100 ppm nitrogen oxides, 180-220 ppm sulfur dioxides, less than 50 ppm unburned hydrocarbons, and water vapor.

Nitrogen makes up a large percent of atmospheric air but is largely incombustible. Atmospheric air is drawn into the boiler to aid in combustion and the nitrogen that remains is vented back to atmosphere. The nitrogen gas does not affect pollution and cannot be used as a measurement for the efficiency of combustion.

Carbon dioxide is the next largest emission in flue gas. High levels of carbon dioxide emissions contribute to greenhouse gases and can be hazardous to human health if they are present in large enough quantities. The ability to accurately analyze carbon dioxide concentrations in flue gas is an important part of environmental monitoring.

The final major component of flue gas is oxygen. The measurement of oxygen exiting the flue stacks is a great indicator of combustion efficiency. Reducing excess oxygen causes boilers to operate more efficiently. Conversely, too little oxygen in the combustion process causes incomplete combustion and excess smoke production, increasing overhead costs and pollution levels.

Hydrocarbon exhaust is also a good measurement of combustion effectiveness. Increased levels of hydrocarbon gases indicate incomplete combustion and negatively contribute to the greenhouse effect. Maximizing boiler output and minimizing atmospheric pollution is critical to improving a company's public image, environmental effects, and bottom line.

For combustion and flue gas management, Wasson-ECE configured an Agilent Technologies Gas Chromatograph (GC) with dual thermal conductivity detectors (TCD/TCD). The TCDs detect hydrogen down to 100 ppm, carbon monoxide to 400 ppm, and methane, ethane, ethylene, propane, propylene, propadiene, acetylene, oxygen/argon composite, nitrogen, and carbon dioxide to 200 ppm. The analysis is complete in approximately 30 minutes. The analyzer utilizes a total of five rugged packed columns that are selected using four rotary valves to create clear separations. Gas sample inject valves are used for maximum precision during injection. Backflush to vent techniques were used to avoid column contamination by heavier components and to avoid interference with the analytes of interest. Only one method was required and injection of 5% methane yielded a relative standard deviation of 0.26%. Data received from a Wasson-ECE flue gas analyzer can be trusted and used to create a more efficient combustion process.

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Pressure Control Yields More Repeatable and Accurate Results

In gas chromatography, sampling valves have been shown to have a low dead volume, reduce sample carryover, and have superior repeatability. The one challenge that exists with gas sampling valves is that they operate under the ideal gas law which states that volume, pressure, and temperature must all remain constant to keep moles of sample injected constant. Volume is fixed by the selection of a gas sampling loop. Temperature is easily and tightly controlled using modern oven technologies. Pressure remains the primary variable in consistent sample injection.

Since concentrations of unknown samples are calculated from the known concentrations of a calibration standard, it is critical to inject the same volume of unknown sample as was injected for the calibration standard. Therefore, it is critical that the unknown sample be injected at the same pressure as the calibration standard.

The most widely used and easiest method of ensuring similar injection pressures is to purge your gas sample valve with the calibration standard for at least 15 seconds. Remove the head pressure by turning the calibration standard regulator off and allowing the gas sample loop to equilibrate to atmospheric pressure before injection. This same procedure when used on samples will yield similar pressure and similar injection volumes. Although this method will improve sampling, it is not ideal. Atmospheric pressure fluctuates based on changes in ambient temperature. If the calibration is performed during the warm summer, it could skew the concentrations reported on the unknown samples during the cold winter. Less dramatic atmospheric pressure changes can occur even within the same day altering the quantity of sample injected and the final calculated concentrations.

To maximize your gas chromatograph's performance a means of pressure control during injection and a record of the actual inject pressure is required. Wasson-ECE introduces the Variable Pressure Sampler (VPS) to satisfy these requirements. The Wasson-ECE VPS measures and stores the pressure of the calibration blend at the time of injection. As each subsequent sample is injected, the VPS adjusts the pressure of the sample to match the pressure of the calibration blend. Using an inert gas supply and an external vacuum pump, sample injection pressure can be adjusted between 25 torr and 2300 torr.

Since the volume of a sample injection valve is directly related to pressure, the Wasson-ECE VPS also allows you to use one calibration blend and adjust the volume injected by changing the pressure to create a versatile multipoint calibration curve. This capability decreases overall lab costs by eliminating the need to purchase and store multiple standards at varying concentrations and provides more reliable results than a single point calibration. The multipoint calibration function also allows you to test for signal linearity at different pressures. With this feature you can easily pinpoint the range of pressures where you will receive accurate and meaningful results from your calibration standard. The more variables that are removed from a scientific analysis, the more reliable the data. The Wasson-ECE VPS is a useful analytical tool to remove pressure related variables.

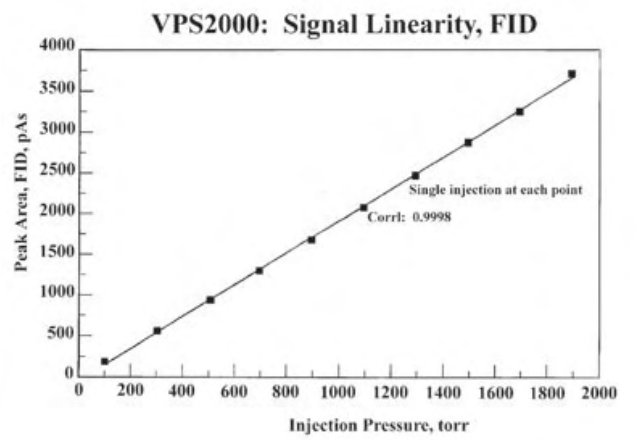

Fig. 1: Example range of linear values for the calibration blend

Chromatography Tips and Tricks

While there are many different types of inlets, the most commonly used inlet is the split/splitless inlet because capillary columns are easily overloaded and perform best when only a small portion of the injected sample is introduced to the capillary column. The split/splitless inlet also allows entire injection to be introduced onto the capillary column for trace analysis when it is in the splitless mode. The versatility and high level of functionality when used with capillary columns make the split/splitless inlet a good choice for most gas chromatograph analysis.

The split/splitless inlet allows the user to alter the chromatography and the method by adjusting the amount of sample introduced to the column by adjusting the split flow. This can change separations, repeatability, carrier gas use, and detectable limits.

The split flow directly represents the ratio of sample that is injected onto the column and the sample that is sent to vent. The higher the split flow, the smaller the amount of sample introduced to the column. Common split ratios range from 1:1 to 500:1. As split flow increases, linear velocity increases and the sample is introduced to the column more rapidly. This rapid introduction causes the analyte band width to be narrower at the column head and although some broadening occurs through the column, the width at the beginning plays a big role in overall peak width. Simply put, higher split flows result in sharper, narrower peaks.

The more rapid sample introduction that is seen in higher split flows also limits the amount of time a sample spends in the inlet, reducing the exposure to high thermal zones that can lead to sample decomposition.

If compounds of interest are co-eluting, another advantage of increased split flows is that by reducing the quantity of sample on the column, you reduce the quantity of each individual compound. In certain cases of co-elution, increasing split flow enough can create clear, individualized peaks.

The negative side to increased split flows is the increased loss of sensitivity. Increased split flows introduce smaller sample sizes causing the sensitivity to decrease and lower detection limits to increase. Loss of sensitivity will also affect repeatability. The higher the split flow the more difficult it is to consistently separate and detect that sample. Increasing split flow will increase the relative standard deviation between runs of the same sample. Increased split flows can also dramatically increase carrier gas usage because extra gas is constantly required to split the sample to vent resulting in increased material costs.

Split/splitless inlets can be a powerful analytical tool as long as the advantages and disadvantages are carefully examined when setting up a chromatography method.

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

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:

- Impurities in Chlorine Gas
- PDMCH in Ambient Air and Feeder Oil
- Impurities in Ammonia
- Determination of Unreacted Monomer in Plastic
- Fluid Inclusion Volatile Analysis
- Analysis of Glutaraldehyde and Acetone in Water

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 20 years of experience can make.

Events Calendar

October 16th-17th: Gulf Coast Conference 2012 in Galveston, TX at Booths # 623 and # 621
March 17th-21st: Pittcon 2013 in Philadelphia, PA

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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