Monthly Newsletter From Wasson-ECE Instrumentation:

Chromatography Corner

ISSUE 09 September 2009

this issue

upcoming events

- Sept. 16-17: GC for Lab Managers Course
  Where: Wasson-ECE in Fort Collins, CO
  Cost: $2,000 per person
- Sept. 23: Free Oxy RGA 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 Trace Sulfurs in Breath Samples by Flame Photometric Detector

Halitosis, or bad breath, is a term used to describe unpleasant odors released when breathing. In most cases, halitosis originates in the mouth. The odors are produced mainly due to the anaerobic breakdown of proteins into individual amino acids, followed by the further breakdown of amino acids to produce foul gases called volatile sulfur compounds (VSCs). VSCs include hydrogen sulfide, carbonyl sulfide, methyl mercaptan, and dimethyl sulfide.

For the analysis of trace sulfurs in breath, Wasson-ECE Instrumentation built a custom sampling system and Agilent Technologies gas chromatograph (GC) modified with dual flame photometric detectors (FPD/FPD).

An FPD works by measuring the light emission of a specific species in a flame. The components of interest are excited to a higher electron state in a hydrogen flame and monitored by a photomultiplier tube (PMT). A filter is used to select the appropriate wavelength of light that reaches the PMT. The lower detection limits (LDL) on the FPDs were 0.1 parts-per-million (ppm) for each component.

A custom vacuum system for automated breath sampling was designed to capture 10 mL of gas sample in less than three seconds and deliver the sample to the GC for analysis.

By customizing an Agilent Technologies GC with dual FPDs, Wasson-ECE was able to separate and quantify VSCs associated with halitosis and assist in the study of bad breath.

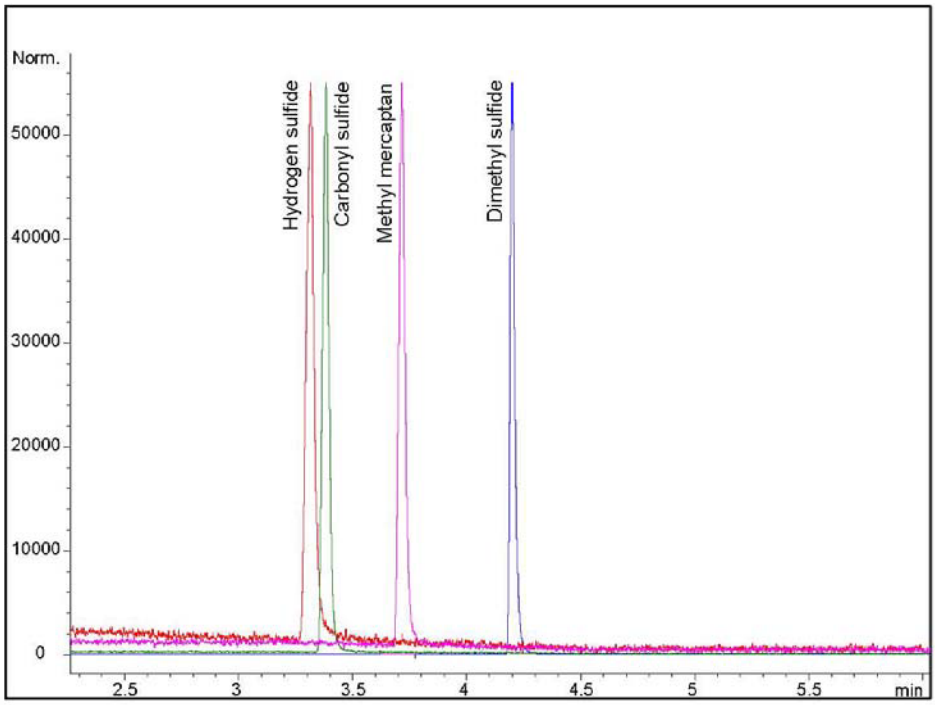

Figure 1: Overlay of four different VSCs chromatograms made by a gas injection on

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Analysis of Gas-to-Liquid Samples by FID/FID/TCD/TCD

Gas-to-liquids (GTL) is a refinery process used to convert natural gas into longer chain hydrocarbons, such as gasoline or diesel fuel using the Fischer Tropsch process. Production begins with the partial oxidation of methane to carbon dioxide, carbon monoxide, hydrogen, and water. Excess carbon dioxide is removed by an aqueous solution of alkanolamine. Carbon oxides must be quantified in GTL gases to make sure the reaction is going to completion and because of their potential as catalyst poisons in later processes.

The Agilent Technologies 7890A series gas chromatograph was configured by Wasson-ECE Instrumentation with dual flame ionization detectors and dual thermal conductivity detectors (FID/FID/TCD/TCD) for the analysis of paraffins, olefins and trace carbon oxides in GTL gases.

Components analyzed on FID A were C_1 through C_5 paraffins and olefins, including methane, ethane, ethylene, propane, propylene, acetylene, isobutane, propadiene, n-butane, trans-2-butene, 1-butene, isobutylene, cis-2-butene, isopentane, n-pentane, 1,3-butadiene, and 1-pentene, with a C_6+ backflush.

FID B analyzed C_6 through C_9 paraffins and alpha-olefins, including n-hexane, 1-hexene, n-heptane, 1-heptene, n-octane, 1-octene, n-nonane, and 1-nonene. Both FID A and FID B had a LDL of 10 ppm. A second method for FID B analyzed trace amounts of carbon monoxide and carbon dioxide using a methanizer to a LDL of 0.1 ppm. The methanizer works by reacting carbon oxides with hydrogen in the presence of a catalyst to produce methane, which is then detected by FID B. TCD A was configured to analyze components including carbon dioxide, ethylene, ethane, acetylene, argon/oxygen composite, nitrogen, methane, and carbon monoxide to a LDL of 400 ppm, while TCD B detects hydrogen to a LDL of 100 ppm. The Wasson-ECE TCD signals were electronically summed to provide a single chromatogram through a single detector signal.

This analysis was challenging due to the large number of components that needed to be analyzed with a single GC system. However, by using dual FIDs and dual TCDs Wasson-ECE was able to separate and quantify paraffins, olefins and trace carbon oxides in GTL gases.

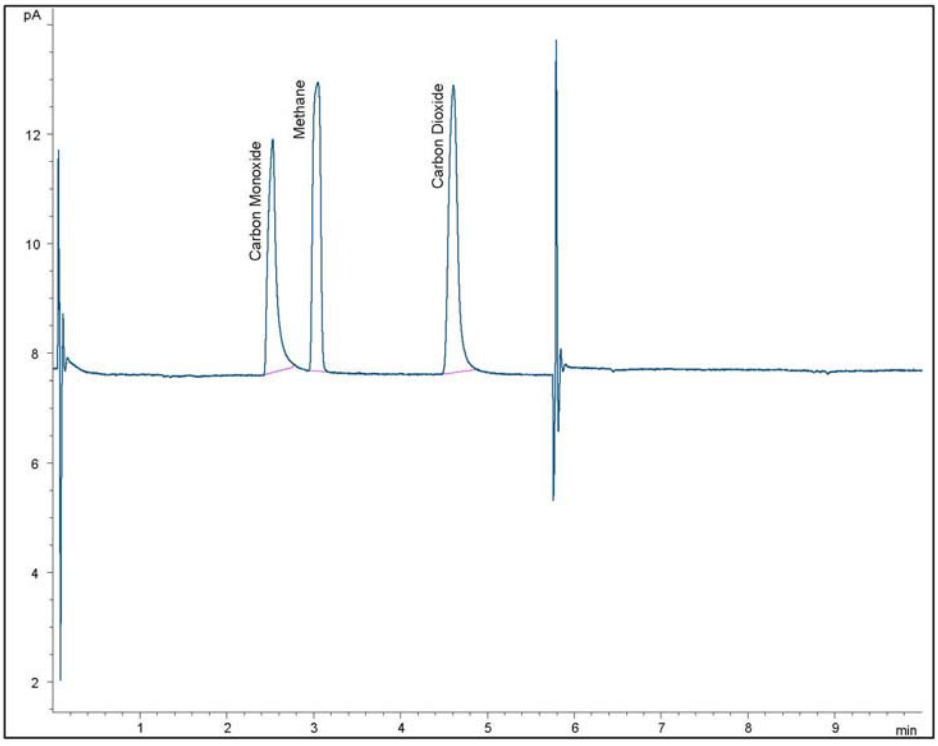

Figure 2: Parts-per-million (ppm) carbon oxides analysis using a 1.0 mL gas valve injection by FID B and methanizer.

Chromatography Tips and Tricks

The air actuator is a rotating piston that has a specific angle of rotation depending on the type of valve. The typical locations of the actuators are behind the auxiliary oven, inside either side panel (if valves are inside the programmable oven) or on top of the GC. Air actuators generally remain maintenance free, but do occasionally run into problems. A leak in the air actuator piston can be mistaken for a leak in the air switch, as the air from the actuator will leak out through the exhaust port of the air switch. As covered in the previous issue of *Chromatography Corner*, when a leak is encountered, a simple test is required to determine whether the air switch or air actuator is leaking. Use the following steps for this determination:

1. Use needle nose pliers to pinch off one of the two air hoses leading to the air actuator at the bottom of the air switch.
2. If the leak stops, the air actuator is responsible for the leak. If the leak continues, the air switch is responsible (refer to *Chromatography Corner* Issue 08 for air switch replacement instructions).

Air Actuator Replacement:

1. To replace the air actuator, turn off the air actuator gas.
2. Cut (do not pull off) the two air lines that lead to the actuator as close to the actuator as possible.
3. Disconnect the actuator from the shaft using a 9/64" Allen head wrench to loosen the collar at the top of the actuator and allow the actuator to slip off the shaft.
4. Replace the actuator and ensure that the rotating pin of the valve (on the valve body) rotates freely between the stops on the body.
5. Turn on the actuator air supply (55 psig).
6. If the pin does not touch both stops, loosen the 9/64" Allen head fitting and allow the actuator to adjust itself correctly to the right position. Do not turn off the actuator air supply.
7. Tighten the fitting again.
8. Repeat the process as necessary.

When replacement of a valve actuator is required, contact Wasson-ECE for the correct actuator. Note: the "T" added to the valve part number refers to a high temperature actuator used in applications where the temperature may reach 150°C.

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

Question of the Month

If a variable temperature program is used rather than an isothermal temperature program (with the same starting temperature), what parameter will not be affected?

A. Order of components
B. Retention time
C. Peak area

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

September 16-17: GC for Lab Managers Course at Wasson-ECE in Fort Collins, CO
September 30: Free Oxy RGA Webinar
October 14-15: Basic GC Course at Wasson-ECE in Fort Collins, CO
October 28: Free TO-Clean Webinar
November 11: Free Automator Webinar

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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