Monthly Newsletter From Wasson-ECE Instrumentation:

Chromatography Corner

ISSUE 17 May 2010

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upcoming events

- May 26: Free Dynamic Blender Webinar
  Time: 9:00 am MDT
- June 16-17: Basic GC Course
  Where: Los Angeles, CA
  Cost: $1,000 per person

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

Analysis of Compounds in Consumer Aerosol Propellant Products by GC

Consumer aerosol propellant products are composed of a mixture of volatile hydrocarbons, oxides, and refrigerants. Whether intentional or not, releases of volatile organic compounds (VOCs) may affect the environment and human health, depending on the particular chemicals involved, the quantities, concentrations, and the relative locations of "receptors" that are sensitive to the particular chemical. Due to stringent government regulations for VOC monitoring, the composition of aerosol products must be quantified.

For the analysis of consumer aerosol propellant products, Wasson-ECE configured an Agilent Technologies GC with a thermal conductivity detector (TCD). Components quantified included nitrogen, carbon dioxide, HFC-134a (1,1,1,2-tetrafluroethane), HFC-152a (1,1-difluoroethane), propane, dimethyl ether, isobutane, and n-butane.

A Wasson-ECE Tedlar™ Bag AutoSampler was used for sampling. The Wasson-ECE Tedlar™ Bag AutoSampler allows the user to sample up to sixteen bags. The sampling system includes a vacuum pump and all of the required hardware for operation. The auto sampler is controlled with an Agilent ChemStation sequence editor.

Since Tedlar™ is a semi-permeable material, samples should be analyzed quickly. If there are a large number of bags to analyze the AutoSampler will help with sample throughput, allowing a user to analyze samples within an acceptable amount of time.

Tedlar™ Bag AutoSampler Features:

- 16 available sample points
- Agilent ChemStation control
- Increases reproducibility
- Diaphragm vacuum pump
- 1/4" bulkhead connections
- Optional gas sample valve

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GC/MS Analysis of Impurities in Coal and Biomass Fuel Gas

For the analysis of impurities in coal and biomass fuel gas, Wasson-ECE configured an Agilent Technologies gas chromatograph (GC) with the Agilent inert mass spectroscopy detector, flame ionization detector, and sulfur chemiluminescence detector (MSD/FID/SCD).

Components identified on the FID were C_2 to C_4 paraffins and olefins including ethane, ethylene, propane, propylene, acetylene, isobutane, n-butane, trans-2-butene, 1-butene, isobutylene, and cis-2-butene. The lower detection limit (LDL) for the analysis of C_2 to C_4 paraffins and olefins was 1 part per million (ppm) for all components. For this method a technique referred to as "Deans Switching" was employed. This technique changes flow directions across a column by varying pressures and for this application was used to backflush heavier compounds to vent.

Sulfur components identified during method development included hydrogen sulfide, carbonyl sulfide, methanethiol, ethanethiol, dimethyl sulfide, carbon disulfide, thiophene, and benzothiophene by SCD. All the sulfur components quantified on the SCD have a LDL of 10 parts per billion (ppb).

The MSD identified aromatic and poly-cyclic aromatic compounds including benzene, toluene, ethylbenzene, p-, m-, and o-xylene, naphthalene, 2-methylnaphthalene, 1-methylnaphthalene, o-cresol, phenol, p-cresol, m-cresol, acenaphthene, acenaphthylene, phenanthrene, and anthracene.

Additionally, some of the sulfur compounds were also identified on the MSD and included methanethiol, carbon disulfide/ethanethiol (separated by target ion), thiophene, and benzothiophene.

By configuring an Agilent Technologies GC with a MSD, FID and SCD as well as employing special chromatography techniques and multiple columns, Wasson-ECE was able to perform the comprehensive analysis of impurities in coal and biomass fuel gas.

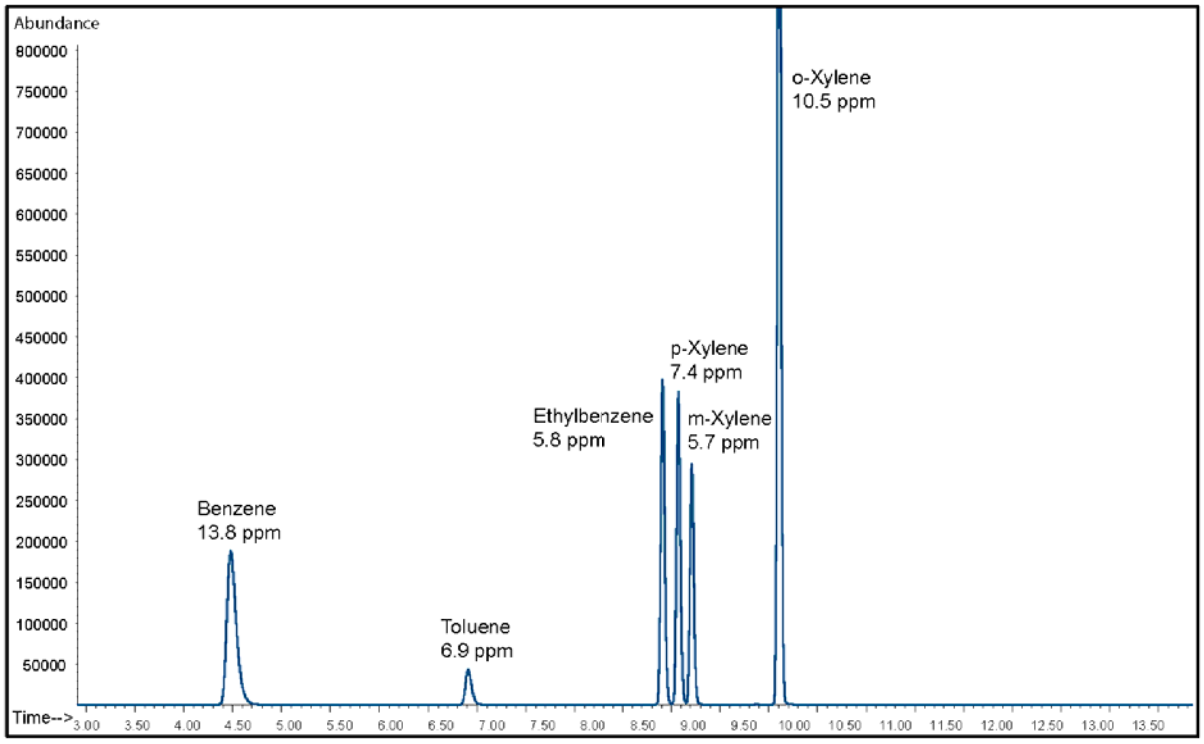

Figure 1: Aromatic compounds by MSD at low ppm levels.

Chromatography Tips and Tricks

The pulse discharge helium ionization detector (PDHID) is a universal, highly sensitive detector. The PDHID has a response to organic compounds over five orders of magnitude with lower detection limits in the single digit ppb level.

Wasson-ECE uses the PDHID for a wide range of low level applications including impurities in permanent gases, ammonia, high purity silanes and monomers. However, because of the sensitive nature of the PDHID it is susceptible to contaminants and high background noise.

The performance of the PDHID is greatly affected by the presence of impurities in the gases used in the GC system (carrier, discharge, and dopant). Wasson-ECE recommends using helium with a purity of 99.999% or greater and always using a helium purifier to remove residual impurities and moisture.

Although brass and copper tubing may be adequate for many GC applications, when working with a PDHID all surfaces that contact the gas stream should be glass or stainless steel and all tubing should be baked-out prior to installation in a PDHID system.

Polymer-based packing in flow controllers or lubricating materials within the valves should be avoided in a PDHID system. Wasson-ECE recommends using purged valves. It is also important to use “ultra pure” grade regulators with stainless steel diaphragms. Neoprene and other elastomers should be avoided.

The background should typically be below 2000 pA. If the system is experiencing a high background signal, the following suggestions may help improve this problem:

1. Check to make sure that your helium purifier (getter) is plugged in and working properly. The sides near the base will be warm if the getter is running.
2. Verify that the flow into the PDHID is correct. The PDHID flow is supplied through a fixed restrictor. The optimum flow is 30 mL/min.
3. Determine if the flow path to the PDHID has been opened recently (i.e. changing a column). If so, then the system may still be purging out the ambient air.

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.

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

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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