Monthly Newsletter From Wasson-ECE Instrumentation:

Chromatography Corner

ISSUE 03 MARCH 2009

this issue

upcoming events

- **March 25:** Variable Pressure Sample System Webinar
  Time: 9:00am MT
- **April 8-9:** DHA Training
  Where: Wasson-ECE in Fort Collins, CO
  Cost: $500 per participant

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

Analysis of Cigarette Smoke Using the Wasson-ECE Cigarette Puffing Machine

The Center for Disease Control (CDC) contacted Wasson-ECE Instrumentation to customize an Agilent 7890A gas chromatograph (GC) configured with an Agilent 5975 MSD for the analysis of toxins in cigarette smoke.

The GC automatically receives smoke from the heated "lung" of the cigarette puffing machine, which is located upstream of a vacuum pump. A diaphragm pump and a multi-position valve are used to simulate the inhaling of smoke by a human lung.

The cigarette smoke is delivered to the GC through a heated bridge to avoid condensation of heavy analytes.

A sixteen position valve is then preloaded and each sample is run sequentially to determine the analyte content of up to sixteen sequential cigarette puffs.

During the analysis the MSD was operated in SIM/Scan mode, collecting both sets of data simultaneously. The SIM mode was used to analyze low concentrations of the desired components, and the Scan mode was used to identify any unidentified components that also eluted in the sample.

The following components were identified during method development to a LDL of 100 parts-per billion (ppb):

- Isoprene
- Acetone
- Acetonitrile
- Acrylonitrile
- 2-Methylfuran
- 2,3-Butanedione
- 2-Butanone
- Propionitrile
- Benzene
- Isobutyronitrile
- 2-Pentanone
- 3-Pentanone
- Toluene
- p-Xylene
- Ethylbenzene
- o-Xylene
- Styrene
- Acetaldehyde

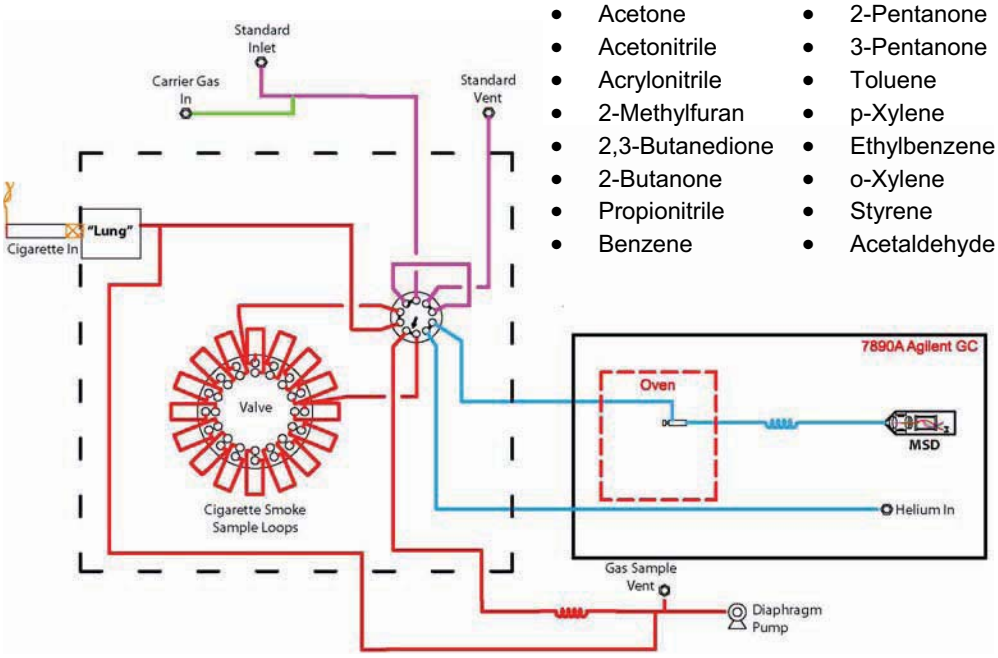

Figure 1: The plumbing diagram shows the Wasson-ECE cigarette puffing machine.

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Determination of Nitriles in MTBE and TAME

Methyl-*tert*-butyl ether (MTBE) and *tert*-amyl methyl ether (TAME) are primarily used as gasoline additives to raise oxygen content and reduce exhaust emissions of volatile organic compounds (VOCs). MTBE and TAME are produced by reacting isobutylene and isoamylene with methanol, using an acid catalyst. Acid catalysts are produced by the hydrolysis of nitriles. Nitriles left in the streams after hydrolysis attack the acid sites on the catalyst which affects MTBE and TAME production.

In the past, nitriles were removed by water washing the product streams. However, water reacts with the acidic sites on the catalyst and forms ammonia (NH3) which also causes catalyst deactivation. For this reason nitriles are removed during fluid catalytic cracking (FCC) and within the delayed coker unit (DCU). These processes involve converting long chain hydrocarbon molecules into more valuable compounds. Acetonitrile and propionitrile are removed with the C_4 and C_5 cuts during FCC and DCU.

Because nitrile removal by FCC and DCU is not always 100 percent effective, propionitrile and acetonitrile must be quantified by GC to ensure the purity of MTBE and TAME.

For this selective analysis Wasson-ECE configured an Agilent Technologies 7890A Gas Chromatograph with a nitrogen chemiluminescence detector (NCD) to detect acetonitrile and propionitrile to a lower-detection-limit (LDL) of 50 ppb. The NCD is nitrogen specific and produces a linear and eqimolar response to nitrogen compounds, enabling complex sample matrices, such as MTBE and TAME to be easily analyzed.

By quantifying nitriles in MTBE and TAME streams, the purity of the acid catalyst can be quantified leading to higher conversions and improved yields.

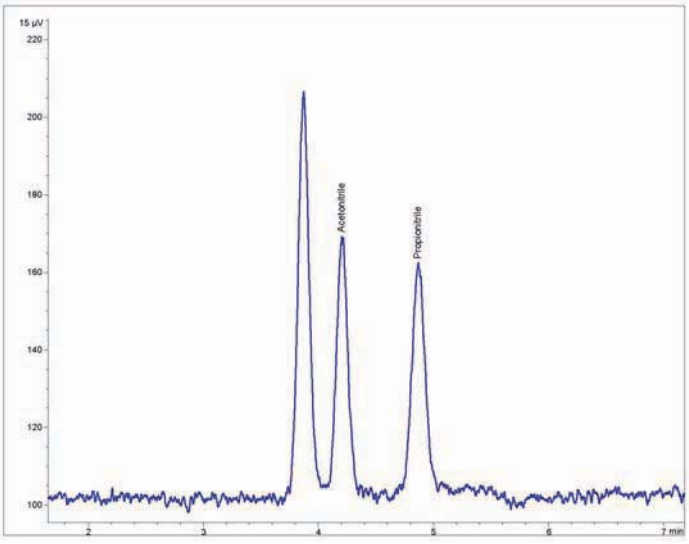


Figure 2: 2.5 uL liquid injection of 100 ppb acetonitrile and 100 ppb propionitrile diluted in MTBE

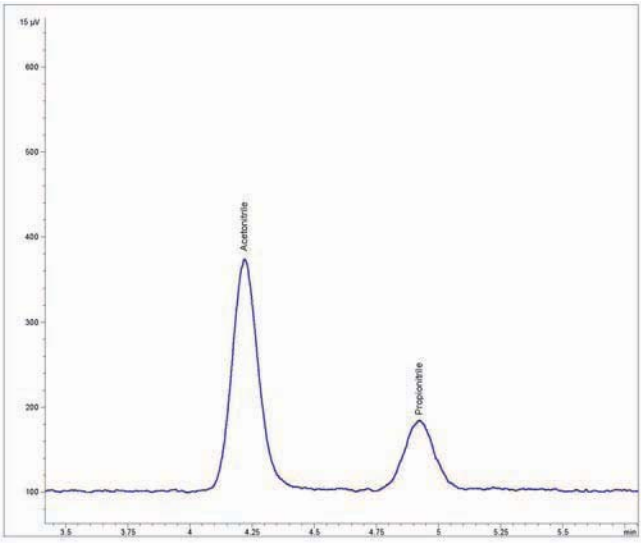


Figure 3: 2.5 uL liquid injection of 333 ppb acetonitrile and 333 ppb propionitrile diluted in TAME

Chromatography Tips and Tricks

Choosing the correct column is critical for analysis by gas chromatography. With so many column choices it can feel like a daunting task. But with a few guidelines, selecting the best column for your analysis can be relatively simple.

When selecting a column, the first decision to make is whether the sample to be analyzed is best suited for a packed or capillary column. Packed columns are able to accommodate a higher sample capacity than capillary columns and for the analysis of gas samples, have the advantage. But for almost all other samples, capillary columns will be much more efficient and provide better peak shape.

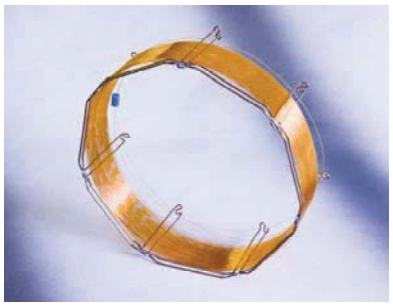
Capillary or packed columns have different variables that must be defined when selecting the appropriate column. For packed columns you will need to define the type of tubing, packing material, mesh of packing and length. For capillary columns you will need to define the type of tubing, the stationary phase and the thickness of that phase (or film thickness). In this article we will focus on open-tubular capillary columns (OTC).

Most capillary column tubing is fused silica, because of its inert properties. The stationary phase can be applied directly to the fused silica or an additional substrate can be added if necessary.

To choose a stationary phase, it is best to gather as much information as possible about your sample. Nonpolar molecules are generally composed of only carbon and hydrogen. Polar molecules usually also contain nitrogen, oxygen, phosphorus, sulfur or halogens. Once you gather information about your sample, choose a column with a stationary phase that is similar in chemical nature to the sample.

Finally, choose the film thickness and the length of the column. The thinner the film, the sooner the components will elute and the better the peak resolutions. Thin film columns are best suited for samples with high boiling components, as they have less stationary phase in which to be trapped. However, if your sample contains primarily low boiling components, it may be best to go with a thicker film. A 30 meter column is appropriate for most analyses. However, for extremely complex samples a longer column will give better separation, because each component spends increased time in the stationary phase.

Packed or capillary, long or short, choosing the proper column for you analysis is critical to achieving success with your GC.



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

Question of the Month

Q: After reviewing a chromatogram, you realize you will need twice as much resolution between two peaks of interest. Your current column is a capillary column and your sample is a liquid at room temperature. Name two things about your analytical method that could be adjusted to give the desired resolution for your sample.

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.

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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 20 years of experience can make.

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Mar Free Variable ressure ample ystem Webinar
A ril DHA Training at Wasson-ECE in Fort Collins, CO
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ne Free Blender Webinar
l Basic GC Course at Wasson-ECE in Fort Collins, CO
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