Monthly Newsletter From Wasson-ECE Instrumentation:

Chromatography Corner

ISSUE 19 July 2010

this issue

upcoming events

- **July 21:** Free Ambient Air Concentrator System Webinar
  Time: 9:00 am MDT
- **August 11-12:** 2-day Basic GC Training Course
  Where: Baton Rouge, LA
  Cost: $1,000 per participant

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

Analysis Headspace from a Reaction Pot by Online Laboratory GC System

The Agilent Technologies GC was custom configured by Wasson-ECE for the analysis of headspace from a reaction pot. Components analyzed on the FID included: methane, n-butane, methanol, ethanol, acetone, isopropanol (IPA), acetonitrile, n-hexane, ethyl acetate, tetrahydrofuran (THF), cyclohexane, diethoxymethane, triethylamine, n-heptane, toluene, and dimethylformamide to part-per-million levels (ppm).

Prior to each sample run, the process system utilized a pressure transducer to check the carrier gas pressure coming into the system. Should the carrier gas be insufficient, an error message was relayed to the computer.

Detector fuel gases were also checked for appropriate pressure and deviations were relayed to the computer.

To provide an accurate and repeatable analysis, the sampling system must deliver a standard sized aliquot of sample to the GC. The sampling system methodology was carefully designed to perform this task regardless of the nature of the incoming sample stream.

Special valves were employed to avoid cross-contamination between samples or between the environment and the sample stream.

The total analysis time was about 30 minutes.

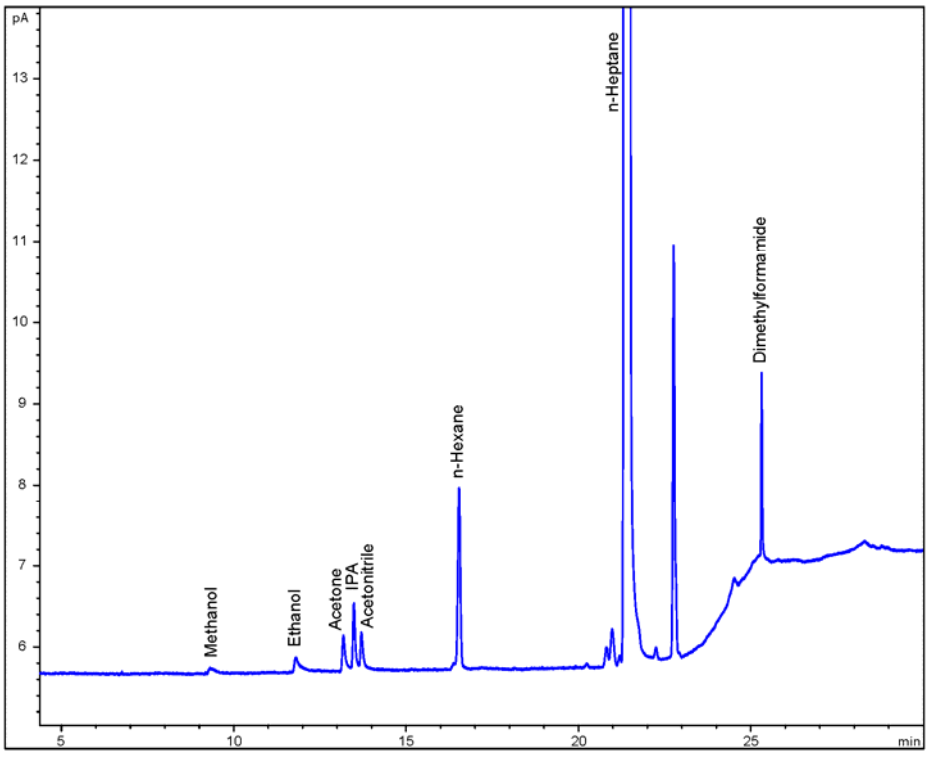

Figure 1: Impurities in the headspace of the reaction pot by FID at 25 ppm.

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Analysis of Components in Paint

Wasson-ECE customized a gas chromatography (GC) and auto-sampler for the analysis of components in paint. With the goal of reducing the ozone formed from aerosol coating product emissions.

The GC was configured with a mass selective detector (MSD). The components are identified and quantified using target and qualifier ions in the paint samples.

The GC uses an Agilent Technologies 7683B Series Automatic Liquid Sampler to introduce samples to the chromatograph columns. The extraction of samples from aerosol containers and the preparation of said samples by the customer was necessary before injection into the GC.

Due to heavy compounds in the samples the system was configured with a pre-column backflush, set to backflush components heavier than n-eicosane (n-C20).

Though n-eicosane elutes at approximately 42 minutes, the analysis is allowed to proceed at an elevated temperature for 50 minutes to ensure all high-boiling components have eluted off of the analytical columns.

Samples run at Wasson-ECE included clear coat, flat white, radiant red and bronze aerosol paints. Below is a chromatogram from the radiant red paint by GC/MSD.

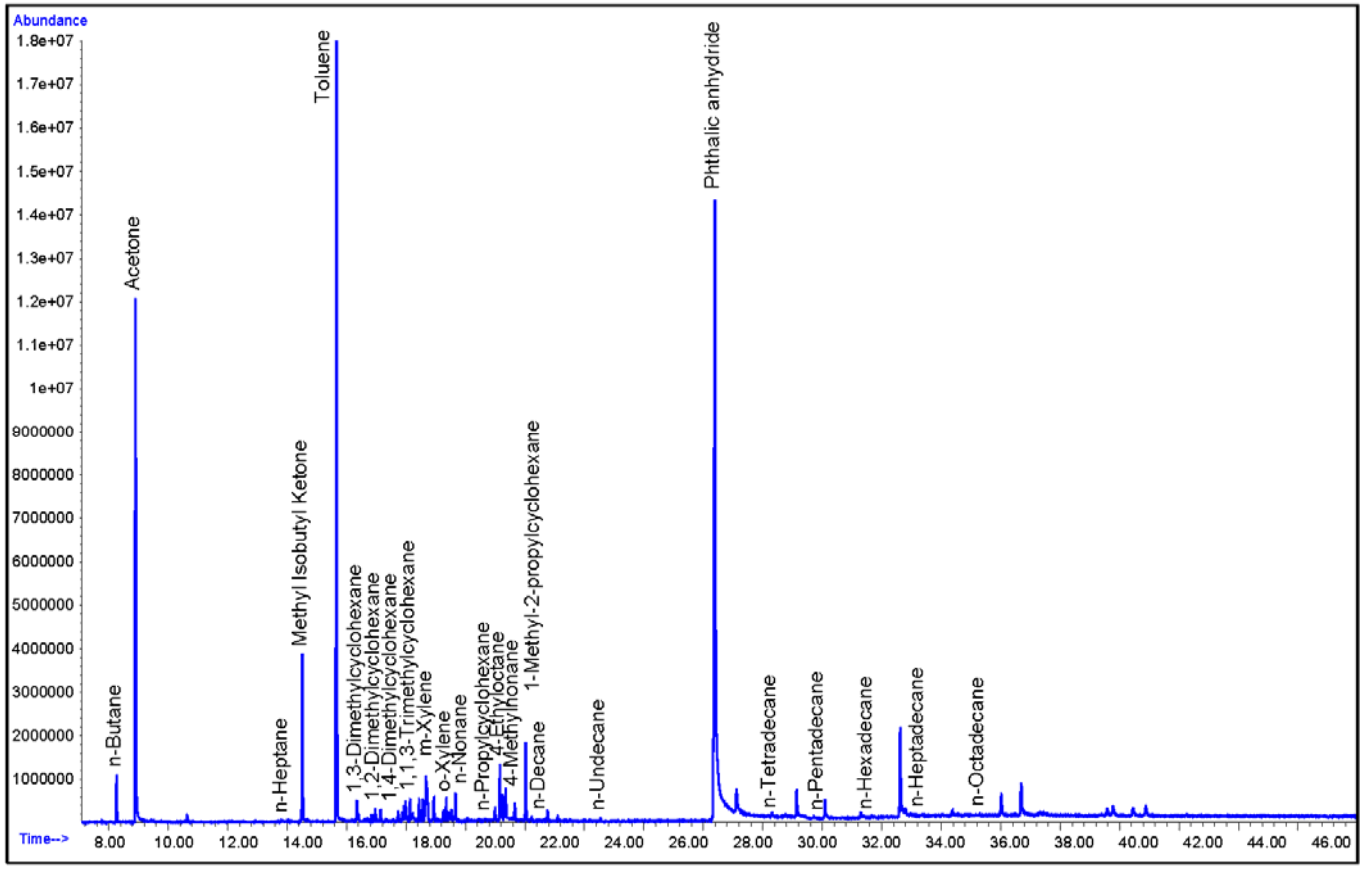


Figure 2: Components found in radiant red paint by GC/MS.

Chromatography Tips and Tricks

Aqueous samples often prove to be very challenging for chromatographers. This article will discuss the most common issues of aqueous injections on GCs.

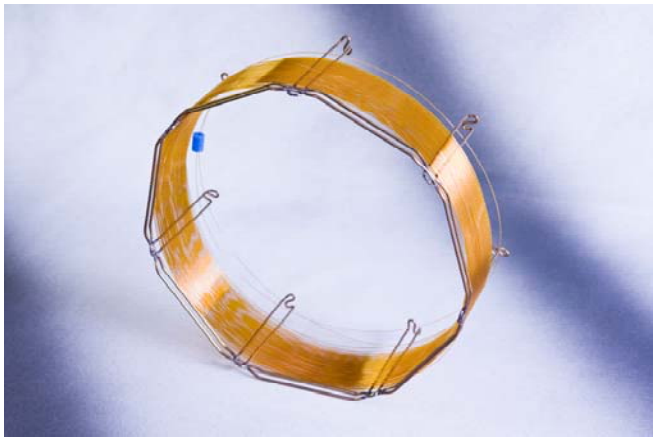
High water samples can cause back-flash in the injector due to the large expansion volume of water. The following list provides some recommendations and precautions to minimize back-flash:

1. Use a large volume liner
2. Choose the smallest injection size possible for your analysis
3. Use a low expansion solvent in your sample
4. Run the injector at the lowest temperature possible for the desired analysis
5. Use high carrier gas flow rates
6. Use high head pressures

In addition to injector problems, aqueous samples can also cause stationary phase degradation in the capillary columns. Capillary columns with a bonded, non-polar stationary phase usually observe little to no change in selectivity, retention, activity or bleed. However, for non-bonded, polar columns, like alumina and molecular sieve, water injections can wash out part of the non-bonded stationary phase which can result in loss of resolution, retention and efficiency.

The amount of stationary phase wash out is dependent on the temperature of the analysis and the solubility of the phase material. If the analysis is at 130°C-200°C, washout is usually minimal. However, at lower temperatures it is much more likely because the water may remain as a liquid rather than vaporizing. Although wash out is gradual, the column lifespan is dependant on the solubility of the phase material. Always observe the manufacturers recommendations.

For all phases, the time to bleed down, or recondition a column after an injection of water is dependent on the run temperature (low temperature injections will take longer than high temperature injections). Wasson-ECE recommends periodic column conditioning or column bake-outs if the high water content sample is injected at a temperature less than 80°C.



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.

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

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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