Monthly Newsletter From Wasson-ECE Instrumentation:

Chromatography Corner

ISSUE 20 August 2010

this issue

upcoming events

- **August 25:** Free Automator Webinar
  When: 9:00 am MDT

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

Determination of Methanol and Ethanol in Denatured Fuel Ethanol

Denatured alcohol is ethanol that has additives to make it poisonous or unpalatable, and thus, undrinkable. Denaturing alcohol does not chemically alter the ethanol molecule. Rather, the ethanol is mixed with other chemicals to form an undrinkable mixture.

Various additives are used to make it difficult to use distillation or other simple processes to reverse the denaturation. Methanol is commonly used, both because of its boiling point being close to that of ethanol and because it is toxic.

Despite its poisonous nature, denatured ethanol is sometimes consumed, which can result in blindness or death if the denatured ethanol contains methanol.

To help prevent this, denatonium is often added to give the substance an extremely bitter flavor. Substances such as pyridine help to give the mixture an unpleasant odor, and emetic agents such as syrup of ipecac may also be included.

Due to strict governmental regulations on blended fuels, the amount of methanol in ethanol must be quantified. Ethanol is required to be between 93-97 mass percent and methanol is required to be 0.1-0.6 mass %.

For the analysis of methanol in ethanol per ASTM D5501, Wasson-ECE customized an Agilent Technologies gas chromatograph (GC) with a flame ionization detector (FID). According to ASTM D5501, the FID will identify methanol and ethanol in denatured fuel ethanol to a lower detection limit (LDL) of 100 ppm.

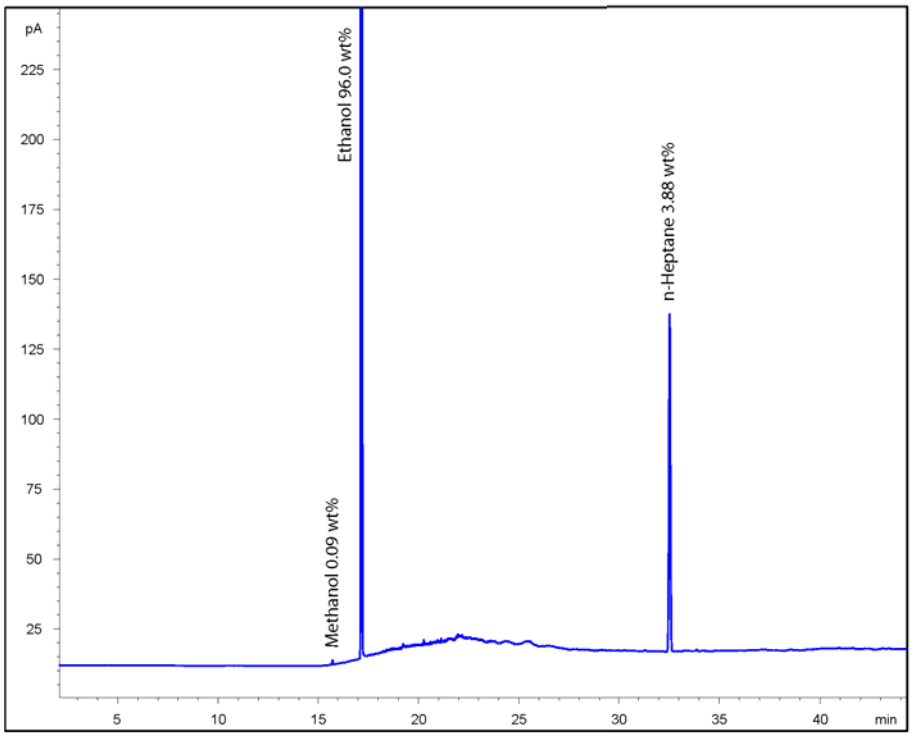

Figure 1: Methanol and n-heptane in ethanol using a 0.2 µL syringe injection on a FID

WASSON·ECE INSTRUMENTATION
Engineered Solutions, Guaranteed Results.

Analysis of N-Methyl Pyrrolidone in Water

N-Methyl pyrrolidone (NMP) is typically used to recover pure hydrocarbons while processing petrochemicals such as the recovery of 1,3-butadiene using NMP as an extractive distillation solvent, and in the desulfurization of gases. Due to its good solvency properties, NMP is used to dissolve a wide range of chemicals, especially in the polymers field. It's also used as a solvent for surface treatment of textiles, resins and metal coated plastics, or as a paint stripper.

NMP has desirable properties such as low volatility, low flammability, and relatively low toxicity. However, it has been identified as a reproductive toxicant. With the increased regulation on NMP, manufacturers are taking extra precautions to make sure employees are not exposed to NMP. This analysis focuses on NMP in water by GC.

Wasson-ECE customized an Agilent Technologies gas chromatograph (GC) with a flame ionization detector (FID) for the analysis of NMP in water. The samples were syringe injected directly to the GC. The lower detection limit (LDL) for NMP was 1 ppm on the FID.

The method used one column to resolve the NMP from other contaminants in the water matrix. The method was established to minimize the analysis and optimize the resolution of the target compound. The analysis was complete after 8 minutes.

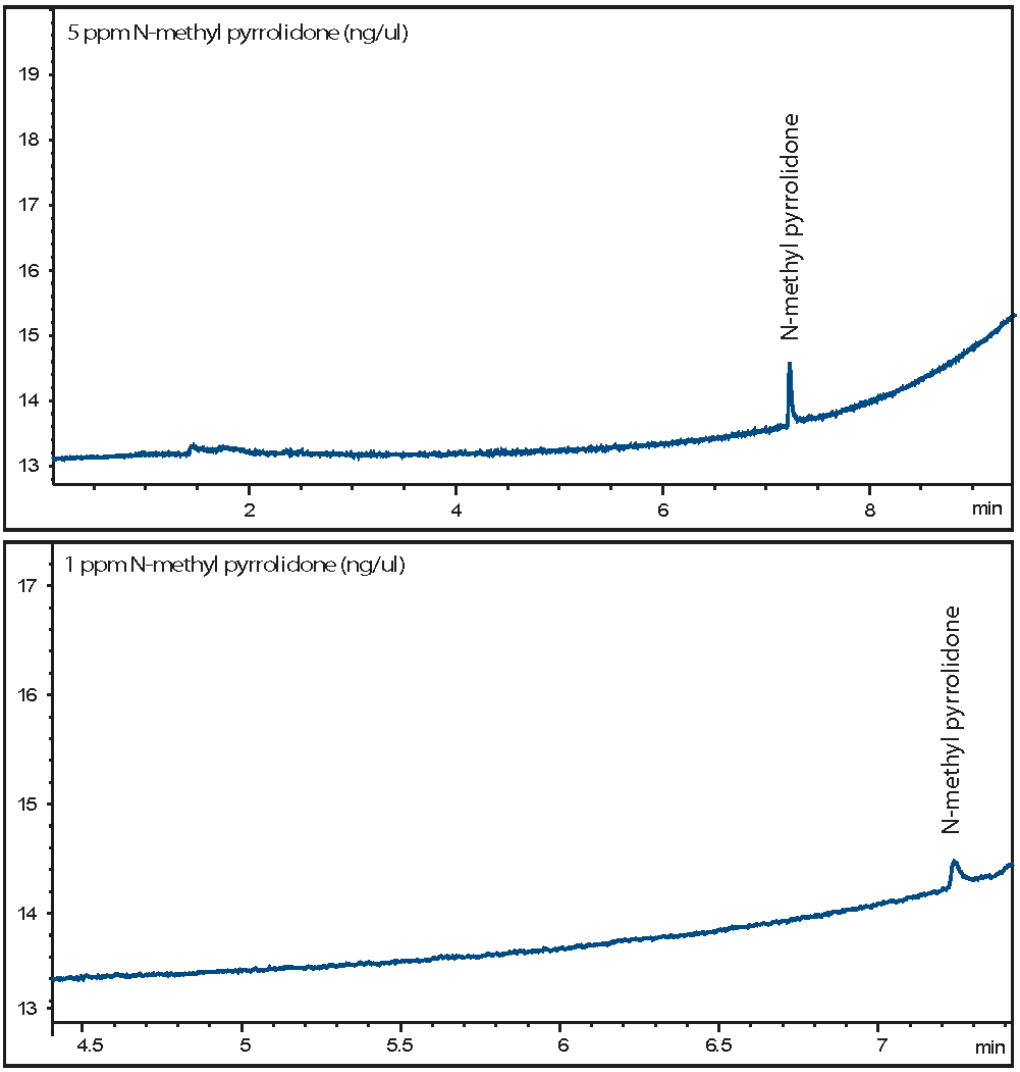

Figure 2 and 3: N-methyl pyrrolidone in water at 5 ppm and 1 ppm by FID.

Chromatography Tips and Tricks

The flame ionization detector (FID) is one of the most widely used detectors in gas chromatography. The FID uses a hydrogen flame to analyze organic impurities or components in samples in the part-per-million (ppm) range. The purpose of this article is to outline a logical procedure for troubleshooting an FID when problems occur.

When using an FID it is imperative that carrier gas purity be 99.9995% or greater and be maintained 20°C greater than the final oven temperature of the GC analysis. FID noise can be caused by factors including gas supply contamination, electrical current leakage, poor flame stability, and mechanical noise from loose FID components.

When troubleshooting an FID the first step is to confirm the integrity of the carrier gas supply. Check gas purities and leak check the plumbing. Also make sure the carrier/ makeup gas is between 80-100 psi, air is at a minimum of 80 psi and hydrogen is at a minimum of 60 psi.

Next, evaluate the level of current leakage in the FID when the flame is extinguished. To test this, turn the FID off and allow the background to stabilize (it should immediately drop and slowly move toward 0 pA). The output should be stable and not jump more than +/- 0.1 pA. If the background stays above 5 pA the problem may be the collector or a loose or contaminated interconnect.

If the FID passes the leak test, next eliminate the column or carrier as the noise source. This can be done by removing the column from the FID and capping the fittings. Re-light the FID and allow it to stabilize. If the FID noise is acceptable the problem may be a contaminated carrier gas or excessive column bleed. If the problem continues measure the FID flows with an electronic flow meter. Measure H_2, air and makeup flows by turning them on one at a time from the GC. They should be within +/- 10% of the set-point. If the flows are significantly off, the jet could be plugged or there could be a leak or defect in the FID pneumatic system.

Once the problem has been identified perform the appropriate maintenance on the FID (clean or replace the jet or the collector), reassemble the FID and make sure there are no leaks in the connections, re-light the FID and bake-out the detector at 350°C for one hour. After the appropriate maintenance and conditioning has been done re-evaluate the FID noise. If problems persist please contact the Wasson-ECE Service Department.

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.

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

August 25: Free Automator Webinar
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.

WASSON·ECE INSTRUMENTATION
Engineered Solutions, Guaranteed Results

101 Rome Court
Fort Collins CO, 80524
www.wasson-ece.com