Monthly Newsletter From Wasson-ECE Instrumentation:

Chromatography Corner

ISSUE 18 June 2010

this issue

upcoming events

- June 23: Free Automator Webinar
  Time: 9:00 am MDT

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

Hydrocarbon Fuel Reforming Analysis

For hydrocarbon fuel reforming analysis Wasson-ECE configured an Agilent Technologies Gas Chromatograph (GC) with a flame ionization and dual thermal conductivity detectors (FID/TCD/TCD).

Two methods were created for the comprehensive analysis. Method 1 was developed on the FID and detected methane, propane, methanol, and ethanol to 10 parts per million (ppm).

Method 2 was developed on Wasson-ECE dual TCDs. Components analyzed included hydrogen (100 ppm), carbon dioxide (200 ppm), oxygen/argon composite (200 ppm), nitrogen (200 ppm), carbon monoxide (400 ppm), ammonia (400 ppm), and water (400 ppm).

The signals from the dual TCDs were electronically summed to provide a single TCD output and chromatogram. The total analysis time for both methods was eleven minutes.

This analysis was particularly difficult because of the wide range of components that needed to be analyzed at ppm levels. However, by employing three detectors the comprehensive analysis could be completed on a single GC system.

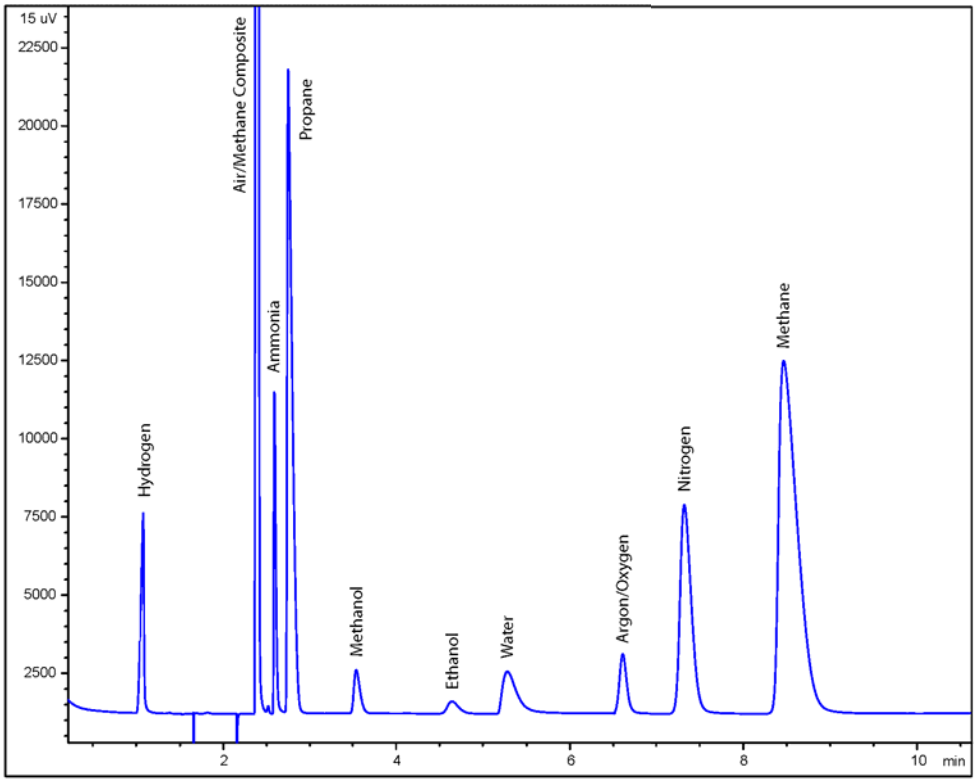

Figure 1: TCD combined analysis without carbon dioxide at ppm levels.

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Process Gas Chromatograph System: Analysis of Reflux and Isomerate

For the on-line analysis of reflux and isomerate, Wasson-ECE customized a three part system. This comprehensive system included an Agilent Technologies GC configured with a flame ionization detector (FID), a sampling system to receive on-line samples, and an on-line computer for unattended control.

To provide an accurate and repeatable analysis, the sampling system had to 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. Samples for this specific system could be delivered at ambient or sub-ambient conditions.

Sample acquisition was facilitated by plumbing sample streams up the on-line GC. The GC system and on-line computer controlled the automated, unattended introduction of sample to the GC.

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

A single method was developed for the detailed hydrocarbon analysis of reflux and isomerate. Components analyzed included ethane, propane, isobutane, n-butane, isopentane, n-pentane, cyclopentane, 2,2-dimethyl-butane, 2,3-dimethyl-butane, 2-methyl-pentane, 3-methyl-pentane, n-hexane, methylcyclopentane, cyclohexane, benzene, 2-methylhexane, 3-methylhexane, 3-ethylpentane, 2,2-dimethylpentane, 2,3-dimethylpentane, 2,4-dimethylpentane, 3,3-dimethylpentane, 2,2,3-trimethylbutane, 1-heptane, ethylcyclopentane, 2,2,4-trimethylpentane, 1,1,2-trimethylcyclopentane, n-octane, ethylcyclohexane, m/p-xylene, n-heptane, methylcyclohexane, dimethylcyclopentane, 1-hexene, and toluene. C_1 through C_4 components co-eluted and were reported as a single peak. The lower detection limit for each component on the FID was 400 ppm.

By utilizing special sampling and computer controls, the analysis of reflux and isomerate was able to be put on-line for greater sample throughput and accuracy.

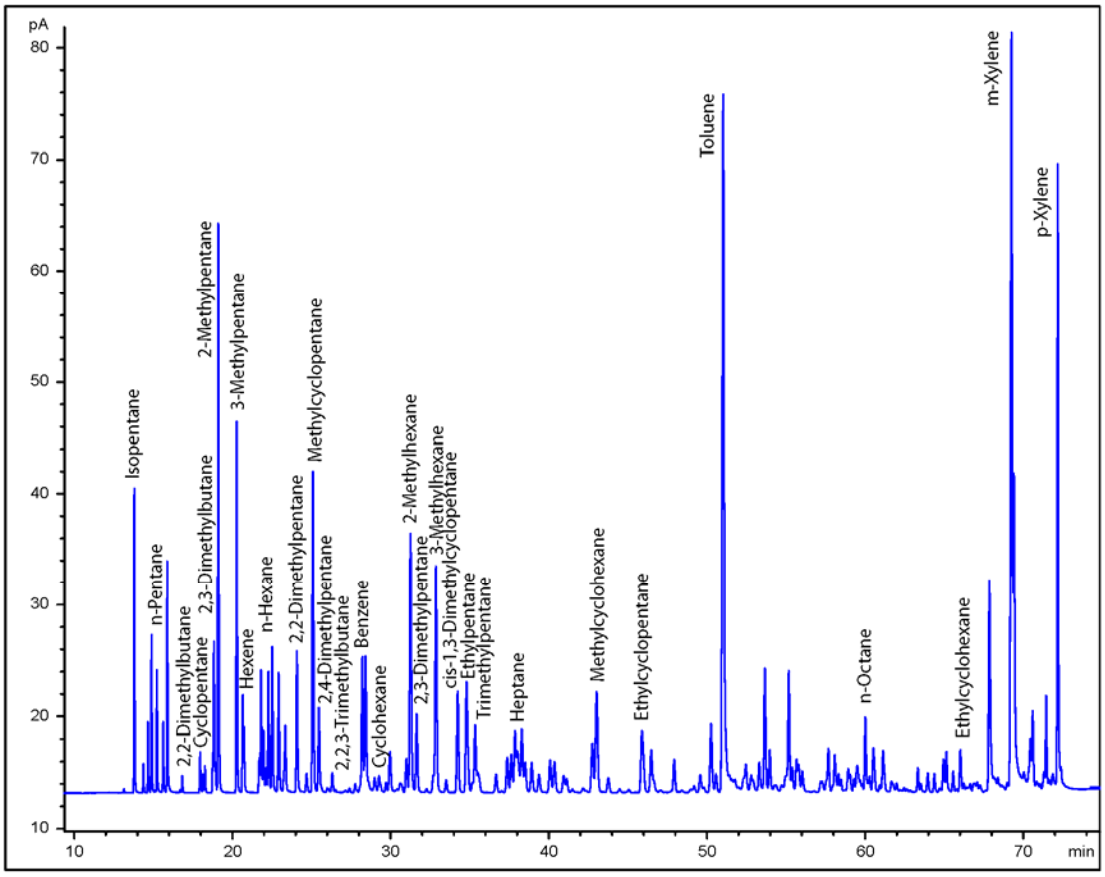

Figure 2: Total 70 minute FID analysis of gasoline at ppm levels.

Chromatography Tips and Tricks

The need for sulfur analyses are increasing as sulfur contaminants in petrochemical products, food and beverage streams, and environmental pollutants are becoming a top priority. As a result, the sulfur chemiluminescence detector is becoming more popular for its ability to achieve very low detection limits along with the ability to speciate sulfur components. The SCD is a complicated detector, but this article will try and break down how this detector works.

The SCD uses oxidation as the main step for detecting the sulfur compounds. The sulfur containing compound is oxidized with air and then reacted with ozone to yield sulfur dioxide and a photon. (see below)

$$R\text{-}S + air \rightarrow SO + H_2O$$

$$SO + O_3 \rightarrow SO_2 + O_2 + h\nu$$

The photon created is detected by a photomultiplier tube and converted to the signal that produces the chromatogram. The process is contained in a partial vacuum to favor the production of the intermediate SO, and not the final product SO_2. Because the sulfur atom is oxidized from the parent molecule, the response is equimolar.

Although this seems fairly simple at first glance, the key point is that these reactions are happening in a sequence. If one portion of this complete reaction is not optimal, then the entire process could be in jeopardy of not providing accurate analytical results. For example, the ratio of air to hydrogen is critical in generating both SO and SO_2 in the correct proportions. Without the correct ratio, the production of SO could be decreased and resulting in low response.

While the SCD is quite complicated it is the only detector available that provides speciation at sub-ppm levels. It is also the only equimolar sulfur specific detector making calibration very simple. Understanding the interworking of the SCD is what leads to accurate analytical results.

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

June 23: Free Automator 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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