Monthly Newsletter From Wasson-ECE Instrumentation:

Chromatography Corner

ISSUE 05 MAY 2009

this issue

upcoming events

- **May 19:** Free Dynamic Blender Webinar
  Time: 9:00 am MT
- **June 24:** Free Automator Webinar
  Time: 9:00am MT

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

Mobile Perfluoronated Tracer Analyzer for the Analysis of Feeder Oil Leaks in High Voltage Feeder Lines

Tracer tagging technology has become an increasingly popular method of finding leaks and tracing flow paths in dielectric fluid filled cables. Wasson-ECE Instrumentation developed a mobile Perfluoronated Tracer (PFT) Analyzer for real time analysis of feeder oil leaks in New York City. PFT was chosen as the tracer to spike cable fluid because it is a non-toxic, non-flammable, water insoluble, chemically stable compound and has a low background concentration of 1-10 femtoliter per liter (fL/L), or parts-per-quadrillion (ppq).

The system utilizes dual ambient temperature sorbent traps to trap and desorb ambient air samples to an Agilent Technologies gas chromatograph (GC) every 90 seconds. The system is able to detect the background ambient concentrations of two PFTs-perfluoromethylcyclohexane (PMCH) and perfluorodimethylcyclohexane (PDMCH) isomers-which are accepted to exist globally from 0.6 to 10 fL/L.

Multi-dimensional chromatography allows the tracer analyzer to be tuned to detect the specific tracer components.

Using heart cutting techniques, the samples are resolved on multiple columns to allow greater levels of specificity. The analyzer is mounted in a mobile lab and samples are taken by driving the system above underground high voltage cables. The system is able to detect feeder leaks by comparing the concentration of the PFT to the background level of PFT. Sampling is fully automated by Wasson-ECE's control software and is interfaced with an electronic GPS map. As the system samples, each sample point is marked on an electronic map denoting the leak status.

By using the PFT Tracer Analyzer, leaks are found in hours instead of days with minimal maintenance and little environmental impact.

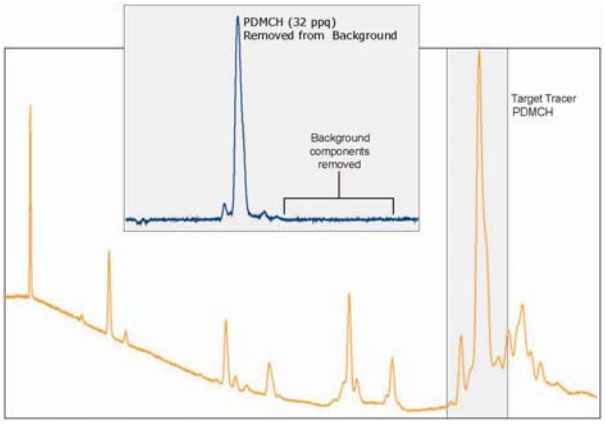

Figure 1: Chromatogram from the PFT Analyzer showing the selectivity of PDMCH from a complex matrix of molecules.

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Analysis of Hydrocarbons in Linear Alpha Olefin Streams

Linear Alpha Olefins (LAO) have the chemical formula C_xH_{2x} and are distinguished from other mono-olefins by the linearity of the hydrocarbon chain and the position of the double bond at the primary position.

The LAO analyzed were produced by the α-Sablin process. The process is a low pressure ethylene oligomerization, using a two-component catalyst system. This system uses a patented zirconium compound and an aluminum alkyl as the co-catalyst to produce a C_4-C_{20}+ distribution of alpha-olefins. Unlike other technologies for the production of LAO, the α-Sablin process is a single step system where chain growth is determined by the ratio of aluminum (Al) and zirconium (Zr).

LAO are used as basic feedstock for polymers, plasticizers, lubricants and detergents. More specifically the intermediate LAO analyzed at Wasson-ECE ranged from C_8-C_{16} and are used for the production of synthetic oils. For the analysis of hydrocarbons in LAO streams, Wasson-ECE configured two Agilent 6890N Series gas chromatographs (GCs) with flame ionization detectors (FID). The GCs were designed to analyze light, intermediate, and heavy (up to octaeiocsene) hydrocarbons in LAO intermediate streams to a lower detection limit (LDL) of 50 parts-per-million (ppm) by weight.

Two GCs provided two temperature programmable ovens to accommodate the wide range of hydrocarbons to be analyzed. The two ovens were each equipped with one FID and were connected together with a heat traced line to provide a single analyzer system with simultaneous separation and quantification. Temperature and pressure effects were used to control the properties of the sample depending on the components of interest. For the analysis of the light components one GC held the pressure under 1150 psig to keep the light hydrocarbons in solution. The second GC kept the sample above 300°C to avoid the condensation of the heavier hydrocarbons. The sample was injected as a pressurized liquid and was vaporized using an on-board vaporizer.

In the past, analysis was done after the separation of the LAO fractions. However, by quantifying the raw product, Wasson-ECE was able to determine the mixture of components before they were fractioned. This process saves time and money by avoiding the initial separation and quantification of the LAO streams.

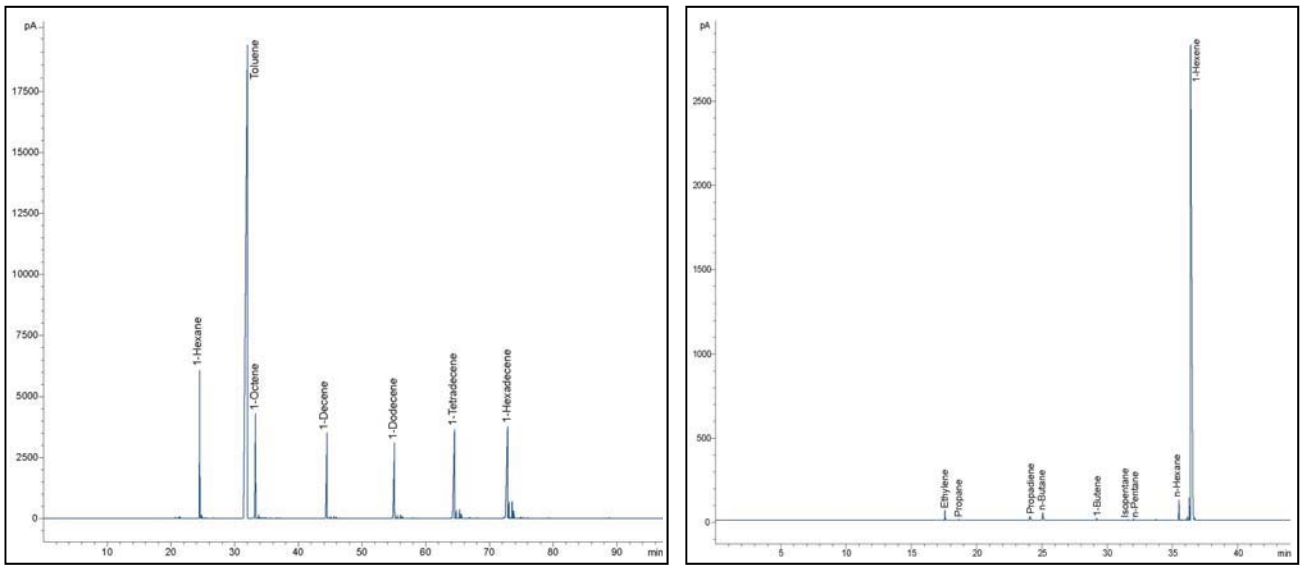

Figure 2 & Figure 3: Chromatograms from GC 1 and 2 showing linear alpha olefins in toluene.

Chromatography Tips and Tricks

The flame ionization detector (FID) is one of the most widely used detectors for analysis by gas chromatography (GC) and uses a hydrogen flame to analyze organic samples. 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 the problem persists contact Wasson-ECE Instrumentation Service Department.

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

Question of the Month

A sample has been introduced using a gas sampling valve on your gas chromatograph. You hear the valve actuate, but as you watch the data handling system, no peaks appear even though the detector is turned on. What is a likely cause for this problem?

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.

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 20 years of experience can make.

Events Calendar

May 19: Free Dynamic Blender Webinar

June 24: Free Automator Webinar

July 29-30: Basic GC Course at Wasson-ECE in Fort Collins, CO

August 26: Free PNA Webinar

September 16-17: Lab Managers Training at Wasson-ECE in Fort Collins, CO

September 23: Free Oxy RGA Webinar

October 21-22: Basic GC Course at Wasson-ECE in Fort Collins, CO

October 28: Free Webinar 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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