Monthly Newsletter From Wasson-ECE Instrumentation:

Chromatography Corner

ISSUE 01 JANUARY 2009

this issue

upcoming events

- **Jan 14:** Dynamic Blender Webinar
  Time: 9:00am MT
- **Jan 28-29:** Basic GC Course
  Where: Wasson-ECE in Fort Collins, CO
  Cost: $2000 per participant

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

On-line Analysis of Chlorosilanes

For the production of polysilicon, which is used in photovoltaic cells, silicon tetrachloride (STC) and dichlorosilane (DCS) impurities need to be quantified by gas chromatography. Chlorosilanes are pyrophoric, making sampling and analyzing difficult. To address this challenge Wasson-ECE built a custom sampling and on-line analysis system using orbital welding, VCR fittings and Hastelloy tubing to ensure a closed inert environment.

Due to the pyrophoric nature of the sample, the most difficult part of the analysis was ensuring that the sample reached the detector without degradation. A key component to achieving this goal was the use of orbital welding. Orbital welding is a process of fusing numerous tubing and component combinations around an arc that is rotated mechanically 360 degrees. By maintaining a perfect balance between gravitational force and surface tension this process avoids inclusions and imperfections in the weld seams.

Along with orbitally welded connections, VCR compression fittings and Hastelloy tubing were used on sample wetted lines. VCR compression fittings ensure a very low leak rate and Hastelloy tubing provides a high resistance to chloride ions, which reduces sample line corrosion.

Once the sample reached the gas chromatograph (GC) for analysis, precautions were taken to protect the switching valves. Prior to injection, the Agilent Technologies 7890A GC system was purged with chromatographic grade nitrogen to avoid the formation of silicon dioxide, which is produced when trichlorosilanes interact with oxygen. The liquid phase sample was then injected and analyzed using a thermal conductivity detector (TCD). The TCD detected silicon tetrachloride (STC), trichlorosilane (TCS) and dichlorosilane (DCS). During analysis, the matrix (TCS) and hydrogen chloride were cut away before reaching the TCD to protect the detector from corrosion. In the field trace chlorosilanes elute from the column to the TCD in the following order: DCS, TSC and STC, with a total analysis time of 12 minutes.

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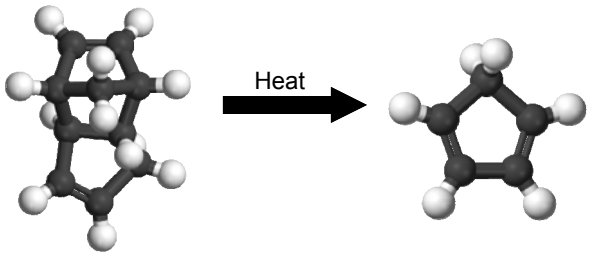
Trace Impurities in Polymer Grade Propylene

Propylene is the raw material for the production of polymer grade propylene, a universal polymer extensively used in several different grades of packaging. All propylene is acquired from petroleum or natural gas deposits during the oil refining process. Because demand often exceeds the supply, most propylene is manufactured by hydrocarbon cracking. The products from cracking contain a mixture of compounds and the propylene is isolated by fractional distillation. One of the impurities in polymer grade propylene is cyclopentadiene. In order to ensure a pure propylene product, trace levels of cyclopentadiene must be quantified

For the analysis of polymer grade propylene, a liquid sample was vaporized and introduced to an Agilent Technologies 7890A GC configured with dual Valco pulsed discharge helium ionization detectors (PDHID/PDHID). One of the PDHIDs was configured to detect cyclopentadiene to a lower detection limit of 10 ppb/mol. The analysis of cyclopentadiene is difficult as it undergoes a Diels-Alder reaction at ambient conditions to form the dimer, dicyclopentadiene.

The original plan to find the retention time of cyclopentadiene was to back-cut and refocus the C_5+ portion. This technique would separate the C_5 through C_9 groups and the results could be interpreted in the following manner: no C_5 indicates an absence of cyclopentadiene in the propylene stream. However, Wasson-ECE found that by cracking the dimer with heat in excess of 300°C to form the monomer, pure cyclopentadiene could be analyzed and the true retention time could be recorded. With the ability to identify cyclopentadiene at low levels, polymer grade propylene producers can assure a more pure product.



Chemistry Lesson: A Diels-Alder reaction is an organic cyclo-addition between a conjugated diene and a dienophile.

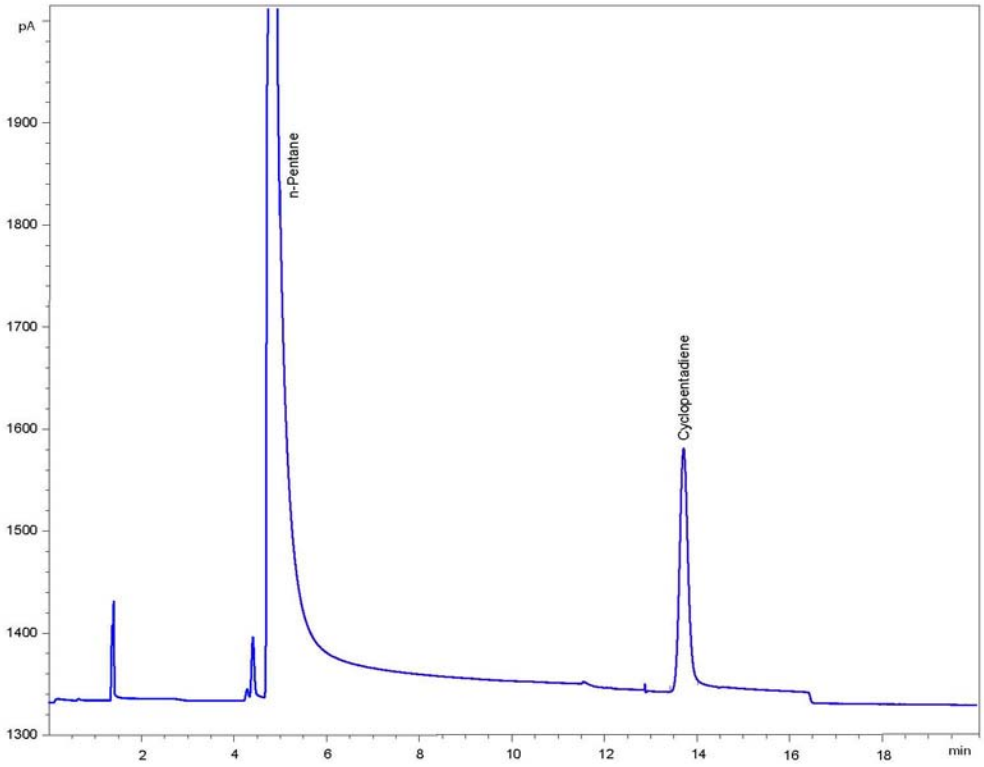
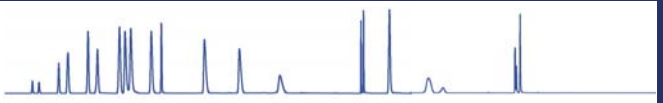


Figure 2: Shows the true retention time of cyclopentadiene in n-Pentane using a 0.25 mL gas sample injection

Chromatography Tips and Tricks

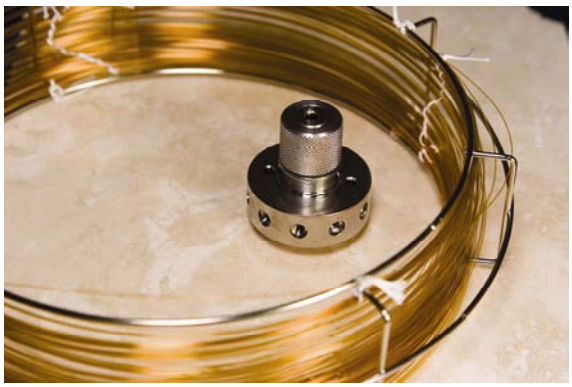


Column contamination is a common problem that leads to invalid data. Unfortunately column contamination is hard to diagnose and mimics other problems in gas chromatography such as a malfunctioning valve or sample line contamination. Some types of contamination may not damage the column permanently, and the columns could therefore be used after the contamination is removed.

There are two main types of column contamination: semi-volatile and nonvolatile. Semi-volatile contamination refers to heavy residual components that build up on a column over time. This type of contamination can result in baseline rise or peak tailing at elevated temperatures. It is important to note that an improperly conditioned column can also give baseline rise as the temperature on the column increases. Semi-volatile contamination can be removed through a process called “baking out”. “Baking out” requires taking the oven, which houses the contaminated column near the maximum column temperature and holding it at this temperature for several hours. For this procedure it is critical to know the maximum temperature of all the components that reside in the oven. If the contaminated column has a higher maximum temperature than another column or component in the oven, it may be necessary to remove the contaminated column and place it into another oven for the “baking out” process. In addition to “baking out”, column contamination can be corrected by cutting off a short length on the front end of the column, since heavy components tend to collect on the front , or inlet side of columns. If neither of the previous procedures work to remove the contamination, the column needs to be replaced.

Nonvolatile contamination refers to residues or components that build up and react with the column phase causing a loss in peak separation or no separation. The only way to correct this type of contamination is to replace the column, because the contaminants have reacted with the column phase and cannot be removed. If the contaminant is consistently in your stream, it may be necessary to add a “stripper column” and backflush capability ahead of the analytical column to prevent future contamination.

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



Question of the Month

Q: Due to poor separation you have decided to replace one of the packed columns on your GC. You remove the old column and replace it with a new one. After ensuring that there are no leaks, you begin to set the flow across the column by setting the pressure. You take note of the previous pressure so that you have an idea of the range for the new pressure. However, in order to get the desired column flow you need to apply 20 times the original pressure. What is wrong with the new packed column ?

Enter for a chance to win a digital camera for your lab. One winner will be chosen randomly from the correct answers. 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

January 14: Free Blender Webinar
January 28-29: Basic GC Course at Wasson-ECE in Fort Collins, CO
February 25: Free PNA Webinar
March 25: Free Variable Pressure Sample System Webinar
April 8-9: DHA Training at Wasson-ECE in Fort Collins, CO
April 22-23: Basic GC Course at Wasson-ECE in Fort Collins, CO
April 29: Free NEW AutoSampler 201 Webinar
May 20: Free LACI Webinar
June 24: Free Blender 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

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