Monthly Newsletter From Wasson-ECE Instrumentation:

Chromatography Corner

ISSUE 11 November 2009

this issue

upcoming events

- November 11: Free Automator Webinar
  Time: 9:00 am MT

Coming soon!
2010 Events Calendar.

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

Analysis of Hexane and Oligomers in Polyethylene by Pyrolysis

During the polymerization process to make polyethylene, hexane is used as a solvent to bring certain components into the reaction. Polyethylene also contains oligomers, which consist of a limited number of monomer units, used to soften the final plastic product.

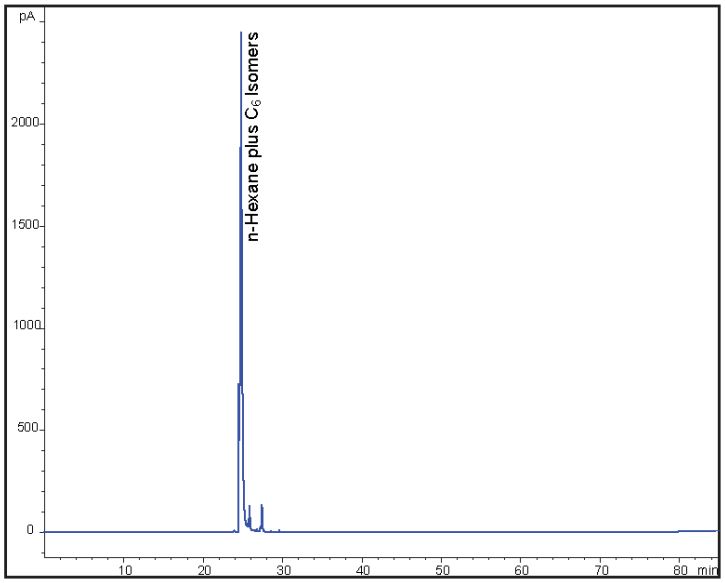
In order to analyze volatile components within a condensed non-volatile sample, pyrolysis gas chromatography (PGC) is used. PGC is a technique that involves rapid heating which decomposes the non-volatile sample and releases volatiles into an inert atmosphere to be quantified by a gas chromatograph (GC).

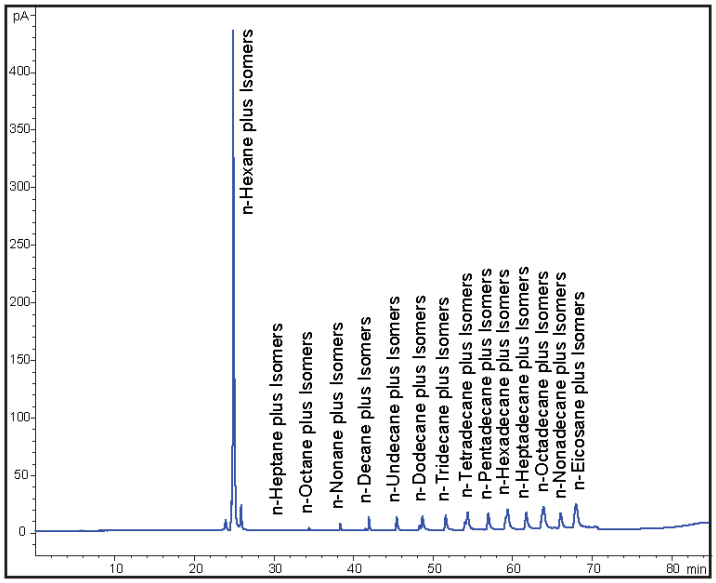
For the analysis of hexane and oligomers in polyethylene, Wasson-ECE configured an Agilent Technologies GC with a flame ionization detector (FID). Two methods were created for this analysis. One for the analysis of hexane in polyethylene and the other for C_7-C_{20} oligomers in polyethylene.

A pyrolyzer was used with ground up polyethylene samples to enable transfer of the analyte materials into the inlet.

The pyrolyzer was mounted over the split/splitless inlet on the top of the GC and was operated by a controller box. Samples were prepared by weighing an appropriate amount of ground polyethylene (approximately 30 mg) in the sample vessel, loading the vessel into the double shot pyrolyzer injector, and injecting the sample into the pyrolyzer burner at the beginning of each run.

By pyrolyzing polyethylene samples, hexane and oligomers within the sample could be released and quantified to a lower detection limit (LDL) of 1 ppm and 20 ppm respectively by GC.





Figures 1 and 2: 30 mg of ground polyethylene spiked with n-hexane and spiked with 100 ppm of C_7-C_{20} n-paraffin standard by pyrolyzer injection and FID.

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Analysis of Trace Acrylonitrile, 1,2-Dichloroethane, and Vinyl Chloride Monomer in Air

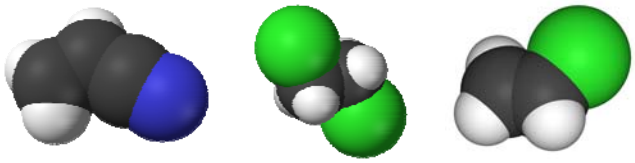
Vinyl chloride monomer (VCM) at ambient pressure and temperature is a gas with a sickly sweet odor, and is highly toxic, flammable and carcinogenic. VCM is a chemical intermediate for polyvinyl chloride (PVC) There are two methods employed for the production of VCM; either hydrochlorination of acrylonitrile or dehydrochlorination of 1,2-dichloroethane. Due to the toxic and carcinogenic properties of these compounds, they must be measured and quantified in workplace air to low part per billion levels (ppb).

Wasson-ECE custom configured an Agilent Technologies gas chromatograph (GC) with dual flame ionization detectors (FID/FID) for the analysis of trace vinyl chloride monomer, 1,2-dichloroethane, and acrylonitrile in ambient air samples.

The three analytes occur in very low concentrations in the ambient air. For this reason, a large air sample is concentrated onto a trap. The sample passes through a multi-bed adsorbent trap retaining the target analytes, allowing primary air constituents (oxygen and nitrogen) to pass through and be vented.

The trap is heated, the analytes desorb, and are then back-flushed onto a split injector before traveling into two parallel columns, each plumbed to a separate detector. Acrylonitrile and 1,2-dichloroethane were quantified to 5 ppb and vinyl chloride monomer was quantified to 10 ppb. Two methods were developed for the analysis of the target compounds. The first method was a “fast method” that resolves vinyl chloride on column 1 and acrylonitrile and 1,2-dichloroethane on column 2. Since this analysis is being performed by FID, a second method was developed for dual column confirmation of the target analytes. The second method is double the runtime of the “fast method”.

By using a concentration system and GC for the analysis of trace acrylonitrile, 1,2-dichloroethane, and vinyl chloride monomer in workplace air, Wasson-ECE was able to quantify low ppb levels on dual FIDs.



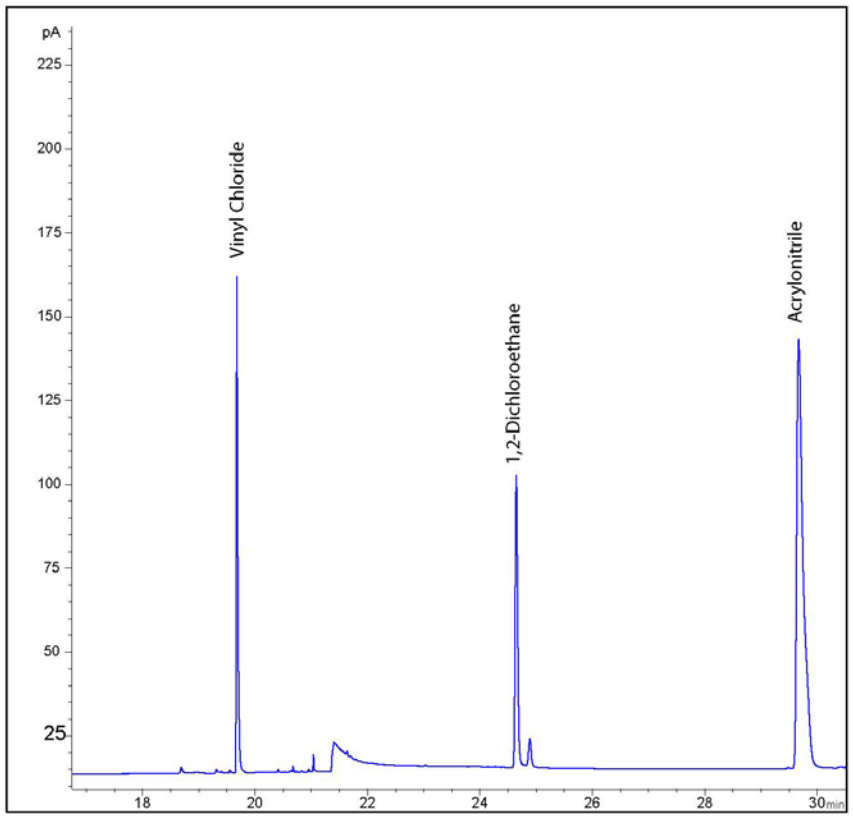


Figure 3: Chromatogram of a blended vinyl chloride, 1,2-dichloroethane and acrylonitrile sample in air by FID A and column 2 at ppbv levels.

Chromatography Tips and Tricks

When installing a new capillary column on an instrument that has been running for a while with set parameters, it is unacceptable for critical analytes to have retention time shifts. However, all columns are not created equal and steps can be taken to alleviate the effects.

Most resolution and retention time shifts are due to small differences in diameter and length of the old and new columns. The previous column was likely trimmed during maintenance so the new column is likely longer than the old one. These small differences affect the actual velocity and flow rate through a column at a given head pressure.

An option for minimizing the effects of column differences is to enter the actual column diameter into the electronic pressure control (EPC) software on the GC. This actual measured value for the diameter is usually reported on the *Performance Summary Sheets* for the column. By inputting the values, the EPC can make adjustments to the flow to compensate for differences in diameter. Remember that if a column is ever trimmed you will want to report the new length to the software as well, so the EPC can make adjustments and compensate.

Flow and velocity are mathematically derived and are based on column dimensions input in the software. Therefore, when a column is changed it is important to make appropriate changes to the dimensions in the software.

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

Question of the Month

A new column does not provide you with the exact calculations for the diameter, which is used in conjunction with the column length to calculate linear velocity (cm/sec). Name another test that could be used to find the linear velocity of the new column.

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 over 20 years of experience can make.

Events Calendar

November 11: Free Automator Webinar

Coming Soon! Wasson-ECE 2010 Events Calendar.

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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