Quarterly Newsletter From Wasson-ECE Instrumentation:

Chromatography Corner

ISSUE 26 October 2011

this issue

upcoming events

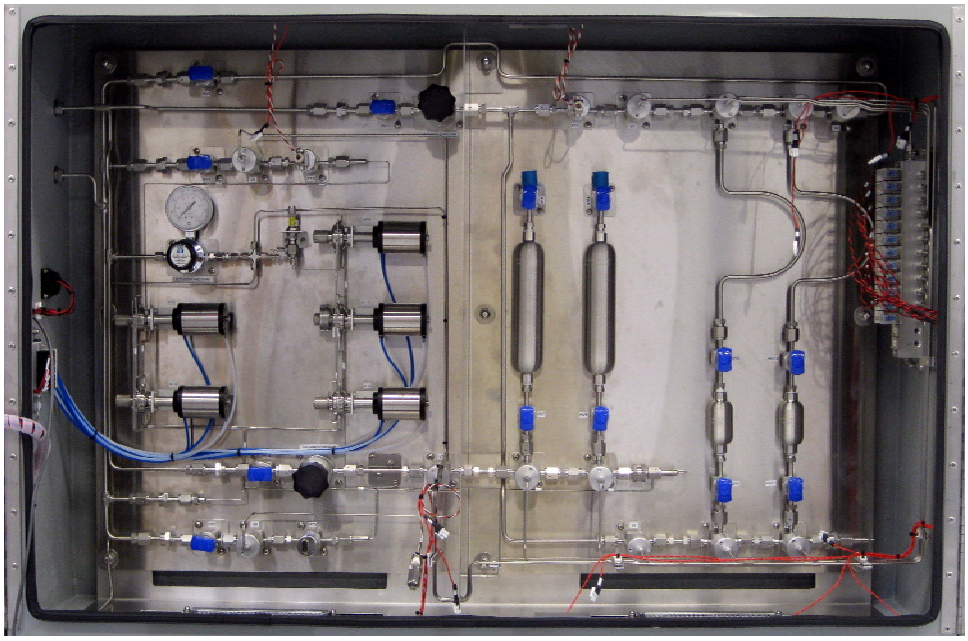
- **Oct 12-13:** Basic GC Course
  Where: Anacortes, WA
- **Nov 30:** Environmental Products Webinar

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

Custom Sample Introduction System for use with Chlorosilanes

The demand for high purity silane gas is increasing due to its use in industries such as semiconductors, LCD displays, and solar panels. One challenge for high purity silane producers is developing the analytical tools to quantify trace impurities. The pyrophoric and reactive nature of silane poses many challenges for this analysis. Special considerations and precautions must be taken to ensure safe sampling, avoid sample decomposition, and prevent atmospheric contamination.

The Wasson-ECE Instrumentation custom sample introduction system is designed to deliver various chlorosilane samples to an Agilent 7890 Gas Chromatograph through four sample cylinders; two gas cylinders and two liquid cylinders.



The system is designed to evacuate residual air from the sample lines that is introduced during the cylinder attachment process. This evacuation process is necessary as chlorosilanes react violently with air to produce particulates and corrosive gas that can damage the sampling system. The sample is evacuated and the system is cleaned post injection as well to ensure safe removal of the sample cylinders. This procedure is simply a series of pressurization and evacuation cycles using an inert gas, such as nitrogen.

The custom sample introduction system included the following hardware:

- Load, acknowledge, and stop buttons
- LEDs to prompt the user
- VCR connections for 50 mL sample cylinders
- Automated and manual valves for sample introduction and purging
- Mounting hardware for installation next to the gas chromatograph
- Fused silica lined sample wetted components

As the industry leader in custom sampling systems, Wasson-ECE can customize a sampling system for many applications and analyses. Contact Wasson-ECE today to discuss your custom sampling needs!

WASSON·ECE INSTRUMENTATION
Engineered Solutions, Guaranteed Results.

Analysis of Trace Ammonia in Ethylene by Nitrogen Chemiluminescence Detection

Worldwide 109 million tons of ethylene are produced each year. Approximately 90% of the ethylene produced is made into various types of polyethylene. Since ammonia is a catalyst poison in the polymerization process it must be quantified to part per billion (ppb) levels.

For the analysis of ammonia in ethylene Wasson-ECE customized an Agilent Technologies 7890 GC with a nitrogen chemiluminescence detector (NCD) to reach a lower detection limit of 20 ppb. The NCD is a nitrogen-specific detector that produces a linear and equimolar response to nitrogen compounds. This is accomplished by using a stainless steel burner to achieve high temperature combustion of nitrogen containing compounds to form nitric oxide (NO). A photomultiplier tube detects the light produced by the subsequent chemiluminescent reaction of NO with ozone. Because of the specificity of the reaction, complex sample matrices can be analyzed with little or no interference. In addition to detecting organic nitrogen compounds, the NCD responds to ammonia, hydrazine, hydrogen cyanide and NO_X.

Features:

- Nitrogen-specific detection for gas chromatography (GC) or supercritical fluid chromatography (SFC)
- Picogram-level detection limits
- Minimal hydrocarbon quenching effects
- Linear, equimolar response to organic nitrogen compounds
- Responds to ammonia, hydrazine, hydrogen cyanide, and NO_X
- Adapters for simultaneous NCD and FID operation

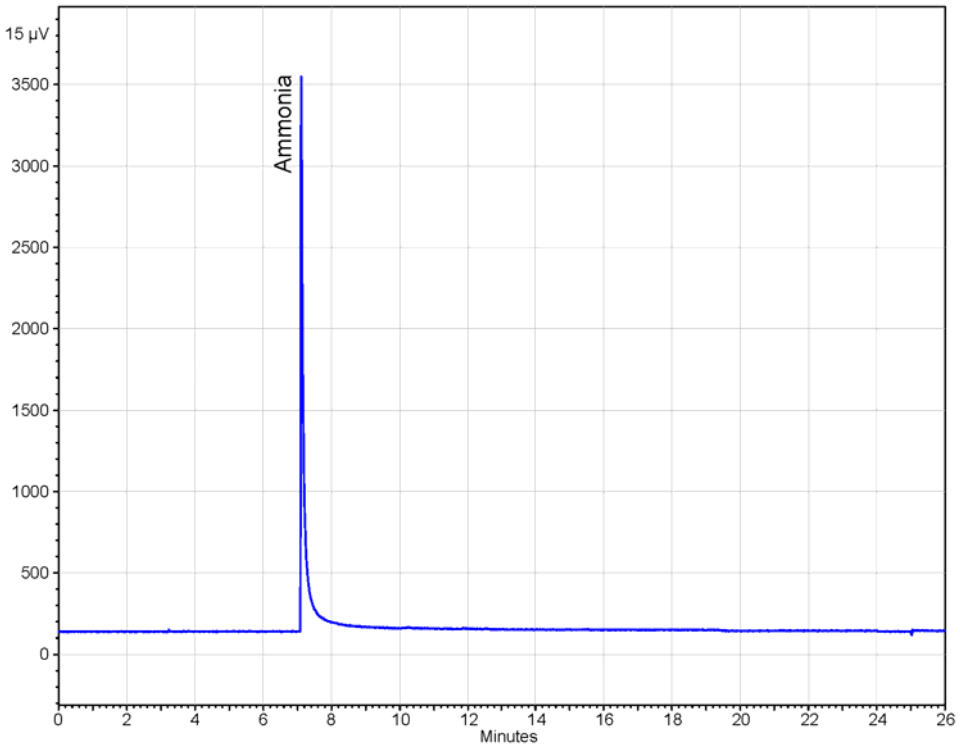


Figure 1: 5 ppm ammonia in nitrogen using a 5 mL sample loop and NCD.

Chromatography Tips and Tricks

If you want to maximize the life of your column, it is important to understand what causes column stationary phases to degrade and what can be done to minimize the effects.

The most common causes of GC column stationary phase degradation are:

- Column contamination from non-volatile and semi-volatile sample residues
- Oxygen damage at elevated temperatures
- Thermal damage by exceeding maximum temperature limits
- Chemical damage from inorganic bases, inorganic acids, and salts

While a decrease in capillary column performance can be attributed to these four causes, the primary cause of failure is contamination from residues in the injected sample. This article will focus on performance tips that minimize this type of damage.

Common symptoms of column contamination are high column bleed resulting in an elevated baseline, peak tailing, and reduced response to active analytes.

Non-volatile contaminants accumulate on the column, and therefore do not elute. The column becomes coated with residues that interfere with active solutes and cause tailing or peak area changes.

Accumulated semi-volatile contaminants might not elute for hours or possibly days. In addition to causing peak shape and area count problems, semi-volatile residues are usually responsible for baseline instability and drift, as well as ghost peaks.

The following tips can be used to maximize the life of your capillary column and minimize column contamination.

1. **Check for residues.** Deposit about 20 µl of sample onto a microscope slide. Set the slide over a heated injection port until dry. If you can see any residue where the sample was, you may potentially be shortening the life of your column with damaging sample residues.
2. **Use a guard column.** Guard columns protect your analytical column from contamination. Sample residues are deposited on the guard column without coating the stationary phase of the analytical column. Guard columns are usually 5-10 meters to allow for substantial trimming before the entire guard column has to be replaced.
3. **Limit bake-out times.** High temperatures over long periods may convert some residues into insoluble materials that shorten column life. Limit bake-out routines to 1-2 hours at an isothermal temperature below the limit of the column.

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

Wasson-ECE Instrumentation News

Wasson-ECE Basic GC 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 12-13: Anacortes, WA

Cost: $1,000 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

October 12-13: Basic GC 2-Day Course in Anacortes, WA

November 30: Free Environmental Products Webinar

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