Monthly Newsletter From Wasson-ECE Instrumentation:

# Chromatography Corner

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## upcoming events

Coming Soon!
2011 Events Calendar.

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## Analysis of Impurities in Styrene

Styrene, also known as vinyl benzene, is an organic compound with the chemical formula $C_6H_5CH=CH_2$. This cyclic hydrocarbon is a colorless oily liquid that evaporates easily and has a sweet smell.

The presence of the vinyl group allows styrene to polymerize. Commercially significant products include polystyrene, styrene-butadiene rubber (SBR) , styrene-butadiene latex, styrene-isoprene-styrene (SIS), styrene-ethylene/butylene-styrene (S-EB-S ), styrene-divinylbenzene (S-DVB), styrene-acrylonitrile resin (SAN) and unsaturated polyesters. These materials are used in rubber, plastic, insulation, fiberglass, pipes, automobile and boat parts, food containers, and carpet backing.

Styrene, most commonly produced from ethylbenzene, which is in turn prepared on a large scale by alkylation of benzene with ethylene.

Wasson-ECE Instrumentation customized an Agilent Technologies gas chromatograph (GC) with dual flame ionization detectors (FID/FID) for the analysis of impurities in styrene.

The dual FIDs identified the following components:

- Methanol
- Benzene
- Toluene
- Ethylbenzene
- m-Xylene
- p-Xylene
- o-Xylene
- Cumene
- Styrene
- t-Butylbenzene
- sec-Butylbenzene
- Diethylbenzene

The components are separated by boiling point and polarity. The lower detection limit is 10 ppm for each component. The total analysis is complete in 45 minutes.

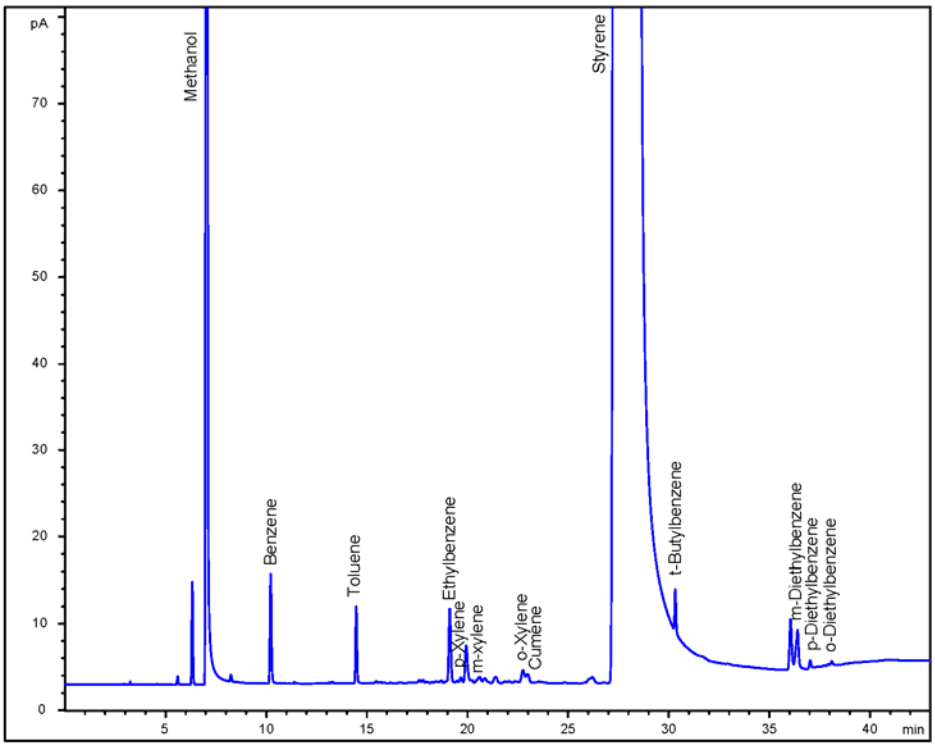


Figure 1: Analysis of impurities in styrene by FID at 10 ppm.

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# VOC Concentration System

The stand-alone concentration system is used in conjunction with a gas chromatograph (GC) or GC/mass spectrometry detector (MSD) for the analysis of low-level volatile organic carbon species (VOCs). The concentrator utilizes a vacuum pump and a mass flow controller, which allows the user to sample from ambient and pressurized sample sources up to 30 psig. Concentration factors of 100 to 500 times are achieved.

The software interface allows the user to enter a load volume between ten and 999 milliliters. An electronic mass flow controller regulates the load volume during sampling onto a sorbent trap, which is running at ambient temperatures. The analytes are drawn onto the trap from ambient air, Tedlar bags, or SUMMA canisters pressurized up to 30 psig. Once the appropriate sample volume is loaded, a dry purge cycle is performed to remove excess moisture and carbon dioxide from the trap. The GC is monitored for a ready status prior to beginning the desorb cycle. When the sample is ready to be transferred to the GC, all flows to the trap are stopped, and the trap is heated to the desorb temperature.

The GC starts when carrier gas is introduced to the trap backflushing the VOCs to the split injector and capillary column. After the VOCs are transferred to the GC for analysis, the bake cycle continues the flow of carrier gas which back-flushes the remaining semi-volatile compounds to a vent located at the back of the concentrator. At the completion of the bake cycle, the trap is cooled to 30 $^{o}$C. A ready light illuminates when the next sample can be loaded.

The next sample can be loaded while the GC continues with the previous VOC analysis. When the sample has completed the dry purge cycle, the VOCs will remain collected on the trap until the GC completes the run and signals that it is ready for the next run. At this point the sample is desorbed to begin the VOC analysis. Thus, higher analytical throughput can be achieved by loading the next sample during the current VOC analysis

The concentrator includes dry purge and bake-out routines to optimize the performance of the analysis.

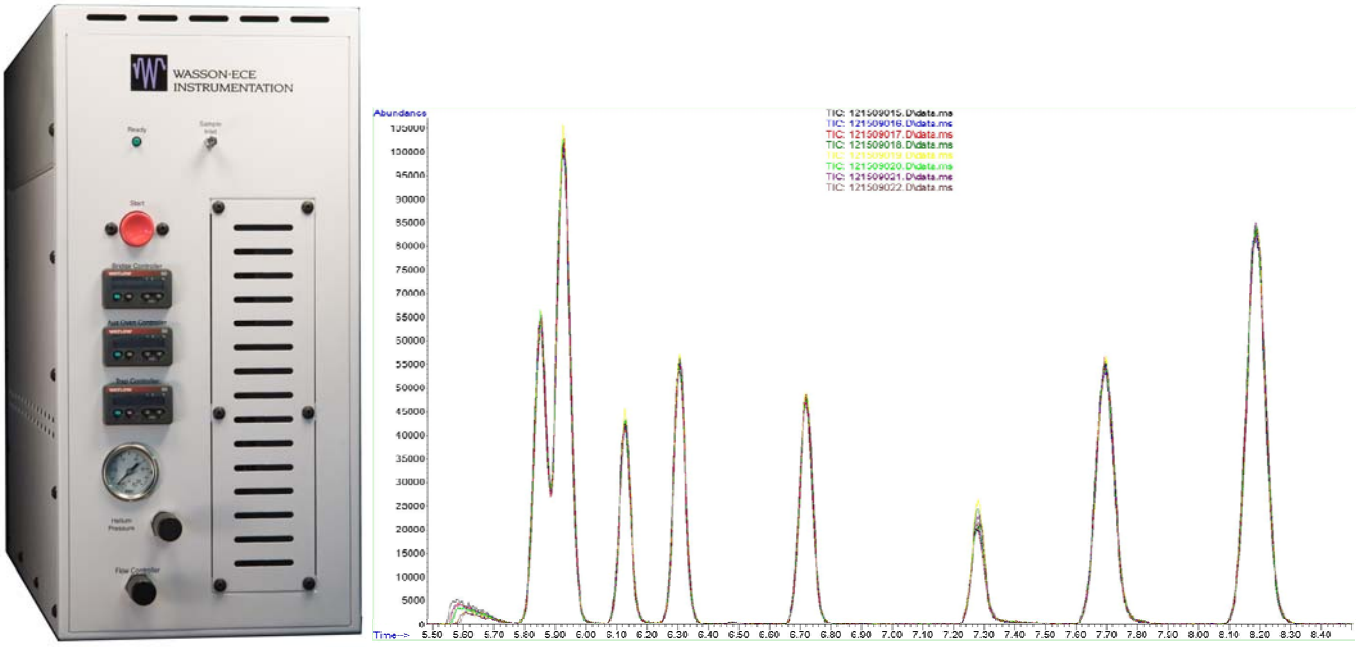


Figure 2: VOC concentration system with repeatability of < 5% on the GC/MSD at 10-999 mL load volumes.

## Chromatography Tips and Tricks

The Nitrogen Chemiluminescence Detector (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. The NCD can be used in the petroleum, chemical, food and beverage, flavor, and environmental industries.

However, due to the sensitive and selective nature of the NCD, it is more prone to chromatographic challenges. This article will discuss common causes of low analyte response on the NCD and corrective actions to ensure minimal GC downtime.

Low analyte response can be due to several issues including improper carrier gas flow, leaks in the detector, or contaminated ceramic tubes.

If analyte response is low, start by checking the hydrogen and air flow rates. If these rates are below the recommended levels in the operation manual, adjusting the rates should alleviate the low response.

Once it has been determined the flow rates are at the appropriate levels and analyte response has not increased, move on to troubleshooting a leak in the detector. The leak can be found by checking the pressures on the controller. If the pressures are not as described in the operation manual, locate and repair the leak and check the integrity of the ferrules.

If there does not appear to be a leak, then the ceramic tubes should be inspected. Contamination of the ceramic tubes can result from column bleed, samples containing volatile metal complexes, or large injection of coke forming hydrocarbons. If any samples have been run containing the previously described contaminants, the ceramic tubes should be replaced.

If all these possible solutions have been explored and the analyte response still does not increase, please contact the Wasson-ECE service department at 970-221-9179.


## Wasson-ECE Instrumentation News

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**Coming Soon!**
**2011 Events Calendar.**

## Events Calendar


**Stay tuned for the 2011 Events Calendar!**

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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.

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