Quarterly Newsletter From Wasson-ECE Instrumentation:

Chromatography Corner

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upcoming events

- **March 2-6, 2014:**
  Pittcon
  Booth 4239
  Where: Chicago, IL

For more information visit:
www.wasson-ece.com
or call (970)221-9179

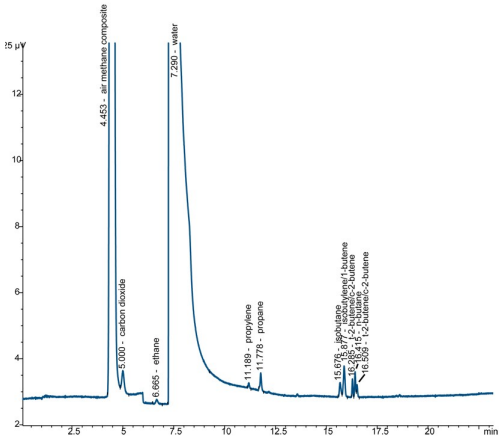
Fluid-Inclusion Volatile Analysis by Gas Chromatography

Fluid inclusions are created when gases or liquids are trapped in a crystal's structure during formation. Fluid inclusions are affected by different temperatures, pressures, and elemental composition of the environment in which the crystal is formed. By crushing crystals to release the inclusion and studying the inclusion composition, valuable information about historical geography can be gained that can aid in mineral exploration.

Wasson-ECE Instrumentation configured an Agilent Technologies gas chromatograph for analysis of volatile fluid inclusions released from four separate rock crushing samplers. The gas chromatograph was custom configured with a 6-port multi-position valve to automatically select between the different rock crushing samplers.

The fluid-inclusion volatile analyzer was designed to be able to handle samples containing up to 100% water, as long as the water remains in vapor phase. A TCD was used to detect an air composite (oxygen, nitrogen, and carbon monoxide), carbon dioxide, water, carbonyl sulfide, sulfur dioxide, hydrogen sulfide, methane, ethane, ethylene, propane, propylene, acetylene, isobutane, and propadiene to a lower detection limit of 30 ppm. The dual FIDs identified methane, ethane, ethylene, propane, propylene, acetylene, isobutane, propadiene, n-butane, *trans*-2-butene, 1-butene, isobutylene, *cis*-2-butene, neopentane, methyl acetylene, 1,3-butadiene, isopentane, n-pentane, neohexane, n-hexane, n-heptane, n-octane, benzene, xylenes, and ethylbenzene to a lower detection limit of 1 ppm.

Figure 1. Fluid Inclusions by TCD



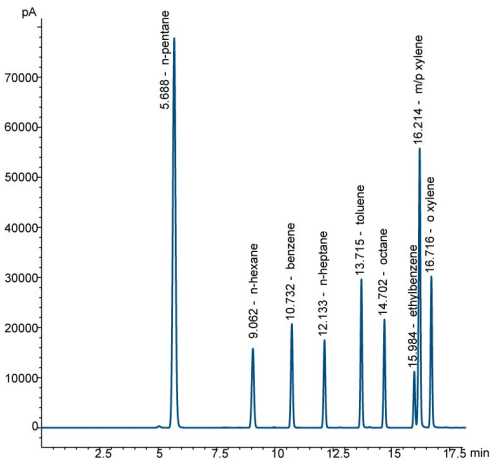


Figure 2. Fluid Inclusions by FID A

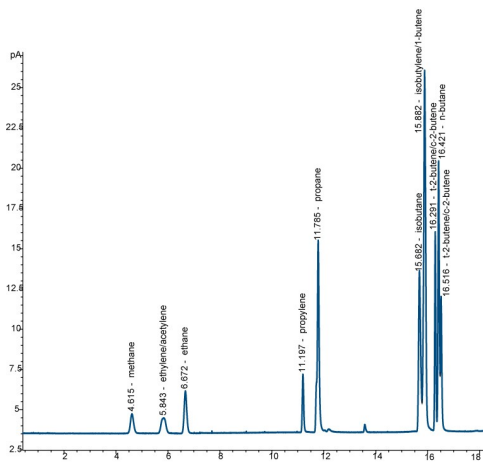


Figure 1. Fluid Inclusions by FID B

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Engineered Solutions, Guaranteed Results.

Vaporizers: Repeatable, Reliable Solution for Liquid Sampling

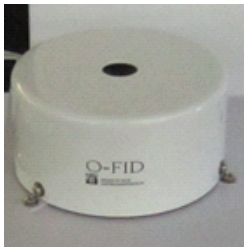
Gas chromatographic analysis of liquefied petroleum gases (LPG's) can be complicated by the high pressures required to keep light, highly volatile components in solution. Injecting these samples as a liquid through a standard liquid sampling valve can result in poor reproducibility of the volatile components. Additionally, the long-term stability of liquid standards can be problematic due to the loss of highly volatile components. Typical vaporizing regulators are inadequate because they cannot flash vaporize a fixed quantity of sample. This results in preferential vaporization of the lower-boiling point components. The large internal volume of a vaporizing regulator requiring extended purge times and the inability to stop the flow also leads to waste of expensive calibration blends.

The Wasson-ECE Vaporizer is designed to allow a very small stream of the liquid to enter a high-temperature zone where the complete sample is flash vaporized. The pressure resulting from this vaporization creates a flow suitable for analysis by gas-phase injection into a gas chromatograph. Since the temperature of the zone is user controlled, complete vaporization of a wide range of sample types is possible and ensures reproducible analyses.

The Wasson-ECE Vaporizer can also be ordered as part of the Wasson-ECE Variable Pressure Sampler for further sampling control.

Run	Ethane	Isobutane	n-C_5+	n-C_7+
1	8562.064	17878.9	31873.9	34391.4
2	8533.369	17571.7	31895.8	35442.0
3	8563.776	17606.4	31976.2	34540.1
4	8538.159	17554.7	31886.3	35549.0
5	8448.108	17628.9	32041.0	35886.2
% RSD	**0.496**	**0.150**	**0.201**	**0.496**

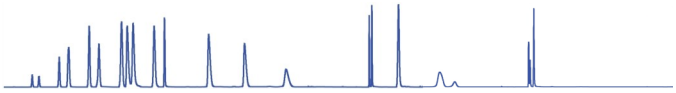
Chromatography Tips and Tricks



The oxygenate flame ionization detector (O-FID) is a selective detector for oxygen containing compounds. In conjunction with a methanizer, the O-FID converts the oxygen into carbon monoxide, then to methane for detection by a flame ionization detector.

The O-FID operates at a temperature of 1250°C and is stored at 750°C. These prolonged high temperatures lead to thermal stress on the O-FID hardware which can cause maintenance difficulties. The majority of the O-FID is constructed of stainless steel and does not degrade at high temperatures. The primary point of concern is the ferrule on the underside of the O-FID that connects the column to the reactor. It is difficult to source ferrules that can withstand the higher temperatures while maintaining a reliable, leak-free seal. Ferrules of varying sizes constructed of graphite/vespel composite and of aluminum materials have all been field tested with varying degrees of success in the past.

Previously, Wasson-ECE had recommended the 0.4 mm ID graphite/vespel composite ferrule (WPN S0320) for use on this connection. This ferrule had provided a tighter, longer seal than larger graphite/vespel composite ferrules and aluminum ferrules. However, at extended exposure to 1250°C temperatures loosening of the connection and leaking was still observed. This required the O-FID to be cooled to allow the connection to be retightened and caused a risk of damage due to the possibility of oxygen exposure to the catalyst.



Recently, Wasson-ECE has conducted further field trials on a 0.5 mm ID graphite ferrule (WPN S0370) and has seen success. The S0370 ferrule provides a more effective seal and does not exhibit the same loosening when exposed to prolonged heat stress. The limitation to the use of the S0370 ferrule is that it can be easily over tightened on installation. Over tightening of the S0370 ferrule will cause the ferrule to extrude up the column and block column flow to the detector.

When installing the S0370 ferrule, the column nut should be finger tightened. The nut should then be gently tightened with a wrench just until snug. Column flow out of the detector must be measured to verify that the nut was not over tightened and the column installation should be observed for leaks using a SNOOP soap solution to verify that the nut has been tightened enough.

The S0320 ferrule is a viable option but after concluding field trials, the Wasson service team recommends the use of the S0370 ferrule on the O-FID column connections for a longer seal at high temperatures. Currently, all new O-FID instruments are being shipped with both ferrules to allow customers to continue to follow their current standard operating procedures or to try the new Wasson-ECE recommended ferrule. For further questions or to replace your current O-FID ferrule, contact the Wasson-ECE service team at (970) 221-91779 or service@wasson-ece.com.

Wasson-ECE Instrumentation News

Wasson-ECE Expands Virtual Application Notes

Wasson-ECE has recently updated our website to include new application notes that highlight analyzer descriptions, chromatography examples, key features and benefits, and additional literature references.

Some of the most recent application notes include:

- Hydrocarbon Impurities in N-Methyl-2-pyrrolidone
- ASTM D7011
- ASTM D7059
- UOP 523

Is there an application you would like to learn more about or see on the website? Email sales@wasson-ece.com 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 30 years of experience can make.

Events Calendar

March 2nd-6th, 2014: Pittcon, Booth: 4239 , Chicago, IL

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.

Happy Holidays and thank you for your business this year!

Wasson-ECE Instrumentation

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