Application Note: 10090

Single Detector Analysis of Refinery Gases

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Key Words

- Cost Savings
- Refinery Gas
- TCD Detector

Introduction

The Refinery Gas Analyzer has been used in refinery laboratories for many years. Refinery Gas Analyzers have traditionally been configured with dual Thermal Conductivity Detectors (TCDs), with one TCD only used to measure Hydrogen. With the increase in sensitivity and performance of TCDs in recent years, it is useful to look at a single TCD instrument to perform the complete analysis. This report describes the performance of a Gas Chromatograph measuring all compo-nents on a single TCD.

Description

There are two considerations which affect the measurement of Hydrogen along with the other components of Refinery Gases on a single TCD. First, the response of Hydrogen when using Helium carrier gas is reduced compared to the Hydrogen response with Argon or Nitrogen carrier gas. This reduction in response is not as great a factor with the increased sensitivity of newer TCD detectors. Second, with Helium carrier, a reversal of thermal conductivity occurs with large Hydrogen concen-trations causing a "W" shaped peak and making accurate measurement impossible (Figure 1).

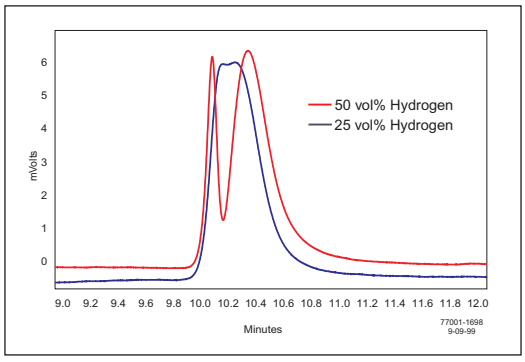

Figure 1: "W" shaped Hydrogen response with high concentrations.

This phenomenon does not occur when the carrier gas is changed to a blend of 8.5 percent Hydrogen in Helium. This report describes the performance of the Finnigan TRACE GC gas chro-matograph configured as a Refinery Gas Analyzer but with a single TCD and using the blended carrier gas. This instrument is configured with an integral valve oven to provide an additional isothermal heated zone for valves and columns. The valve oven is attached to the left side of the instrument to provide easy access to both the valve oven and the program-mable GC oven (Figure 2). The inner heated enclosure of the valve oven is designed to accommodate up to four heated valves, two unheated valves, and 50 feet of 1/8 inch metal columns. The removable cover for the heated enclosure exposes the top and one side of the enclosure. This gives the chromatographer access to all the valve fittings for plumbing or leak-checking (Figure3).

Figure 2: TRACE GC with optional valve oven

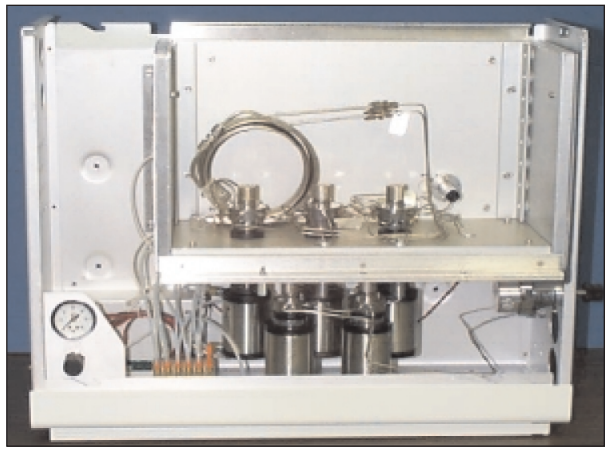

Figure 3: Inside view of valve oven layout.

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The Refinery Gas analysis is an isothermal method, with a backflush of heavier compounds to reduce the method cycle time. By placing the valves and columns for the Refinery Gas analysis in the valve oven, the programmable GC oven is available for other analyses such as Extended Natural Gas or Sulfur Compound distribution.

When the instrument is configured to perform the entire Refinery Gas analysis with a single detector, three valves are used. The gas sampling valve is a 10-port valve which also serves as the backflush valve. The two remaining valves are 6-port valves configured as trap and hold valves for the Molecular Sieve and Porous Polymer columns. These valves are plumbed with variable restrictors to maintain a constant carrier flow rate while the columns are switched into the trap position (Figure 4) .

The Hydrogen peak elutes at approximately 5 minutes, well before the other peaks in the analysis, but after the backflush composite peak. The Hydrogen peak is negative when the blended carrier gas is used, but is changed to a positive peak using the programmable time functions of the TRACE GC. The polarity of the TCD signal is reversed before the Hydrogen peak elutes, and restored immediately afterward. With the blended carrier gas, Hydrogen is a measurable component in all concentrations from less than 0.25 to 100 volume percent. Even though the Hydrogen peak is small relative to the other peaks in the analysis, it is well within the quantitative range of measurement of the data system. Figure 5 illustrates the Hydrogen peak at concentrations from 12 to 100 volume percent. Figure 6 shows the lowest concentrations that were tested. The signal to noise ratio at 0.25 volume percent concentration is excellent, with area counts above 150000.

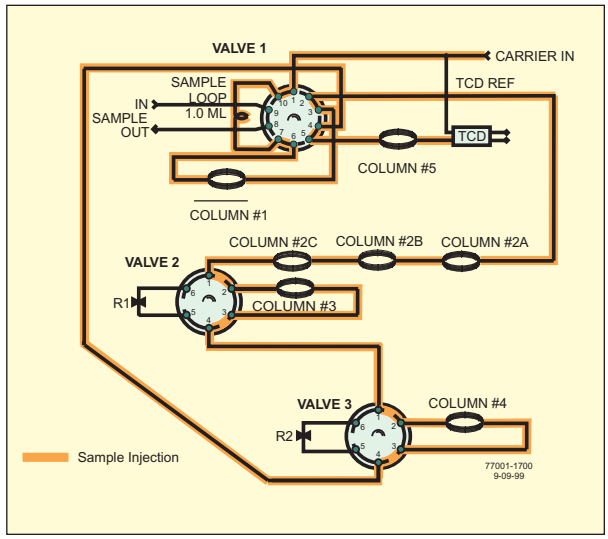

Figure 4: Plumbing diagram showing injection path.

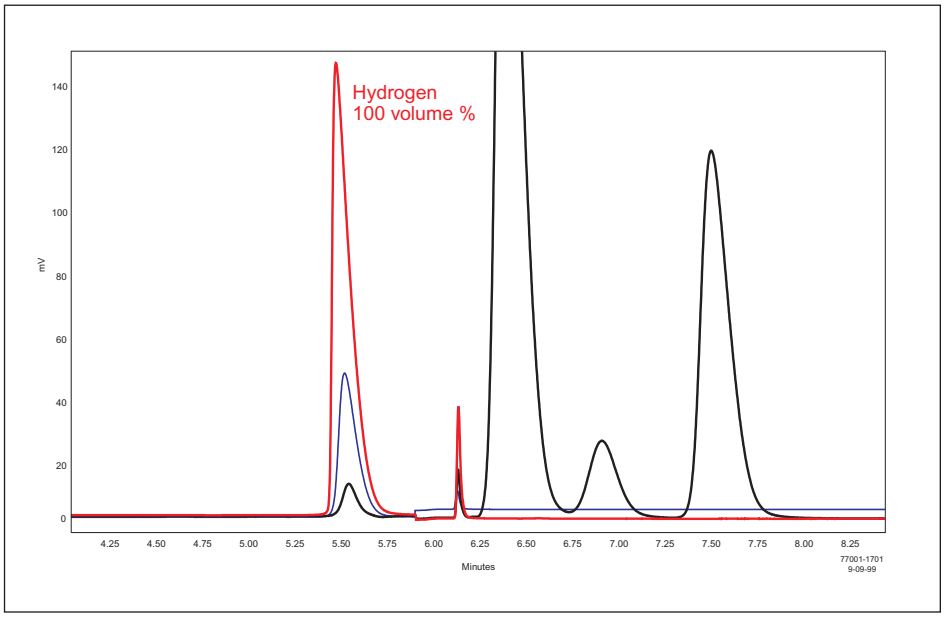

Figure 5: Hydrogen at up to 100 percent concentration

Conclusion

One important advantage to performing this analysis with a single TCD is reduced instrument cost and complexity. With only a single analysis channel and detector, the instrument cost is reduced by the cost of one TCD and its associated valve and columns. This means a cost savings of several thousand dollars compared to the traditional dual detector instrument.

An additional advantage is a simplified method report. All compounds in the analysis are analyzed from a single injection, eliminating the requirement for combining results from two detectors into a single report. The chromatogram shows a complete analysis without detector switching (Figure 7) and results are reported in a single channel format.

Another advantage with this configuration is the ability to switch the instrument between Natural Gas and Refinery Gas Analysis. Natural Gas analysis is usually performed with a single TCD, or as part of a dual channel instrument performing an extended Natural Gas analysis with an FID as a second detector. By configuring an instrument in this way, a single Gas Chromatograph could be used to perform Natural Gas, Natural Gas plus Oxygen/ Nitrogen, Extended Natural Gas, and Refinery Gas analyses with simple column changes.

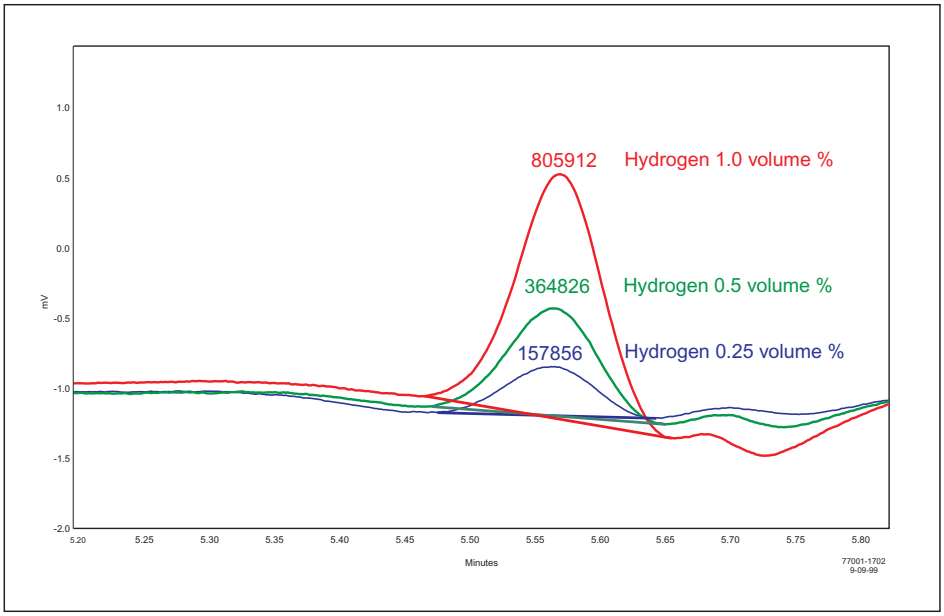

Figure 6: Expanded view of low Hydrogen concentrations.

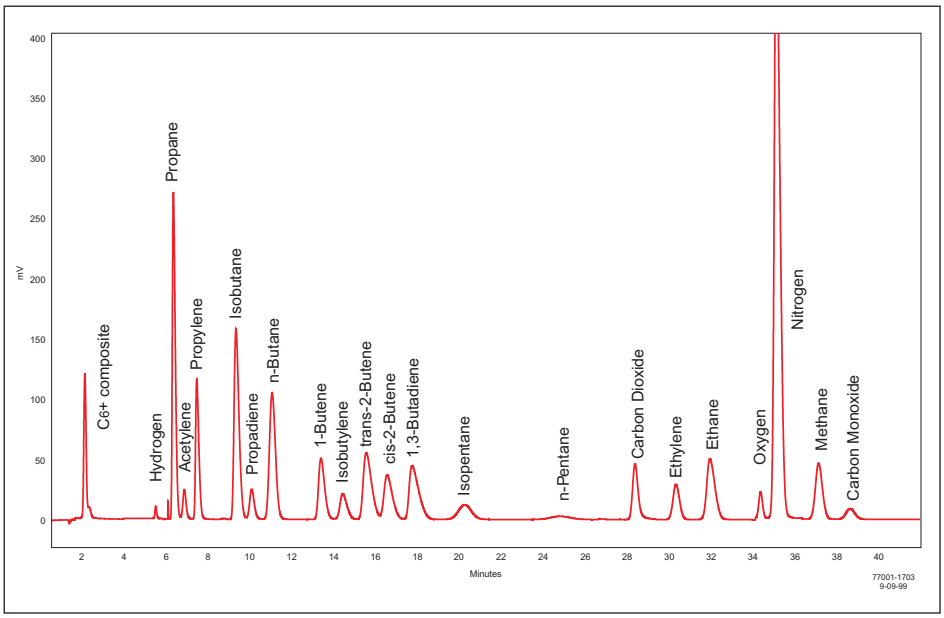

Figure 7: Single detector analysis of Refinery Gas

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