ta3000R Method P-001

Determination of Trace Levels of H_2 and CO in Ethylene and/or Propylene

Introduction

The Trace Analytical™ ta3000R Reduction Gas Analyzer, from AMETEK Process Instruments, is ideally suited for the determination of ppb to ppm levels of H_2 and CO in ethylene and/or propylene streams. Making polyethylene or polypropylene from these gases requires a catalyst, such as the Ziegler-Natta polymerization catalyst. The performance of this catalyst is adversely affected by CO, and it is of interest to determine this component in the ppb levels. In this analysis, the Reduction Gas Detector (RGD) is used to provide rapid and reproducible measurements. Several unique qualities of the RGD enable the quantitation of both H_2 and CO to extremely low concentrations.

Unsaturated hydrocarbons give strong response in the RGD. When ethylene or propylene is a major component in the sample, it can overload the RGD as well as the analytical column, producing a long tailing peak that requires a long time to return to baseline. This application package is designed to overcome interference from the major unsaturated component by special plumbing and column configuration.

The Trace Analytical Method

The flow diagram for method P-001 is shown in Figure 1. Nitrogen carrier gas is used because it gives a minimum of upset in the baseline from the injection, is inexpensive and readily available.

A 10-port valve is used to inject sample from the sample loop to the two stripper columns 1A and 1B. Lighter compounds such as H_2 and CO elute quickly from stripper columns into analytical column 2, while higher molecular weight components such as hydrocarbons (principally ethylene and propylene) and moisture remain in the stripper columns. After the light components have had sufficient time to elute from the stripper columns into the analytical column, the 10-port valve returns to the load position. In addition to preparing the analyzer for the next sample injection, this valve position is used to back flush the higher molecular weight components from the stripper columns to vent.

Back flushing of the stripper columns to vent accomplishes two objectives. It shortens the analysis time and it vents contaminants which can produce an unstable baseline away from the detector.
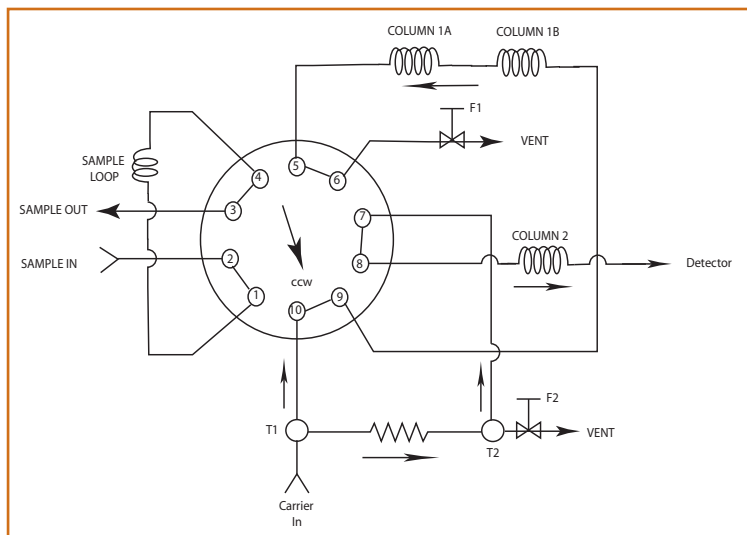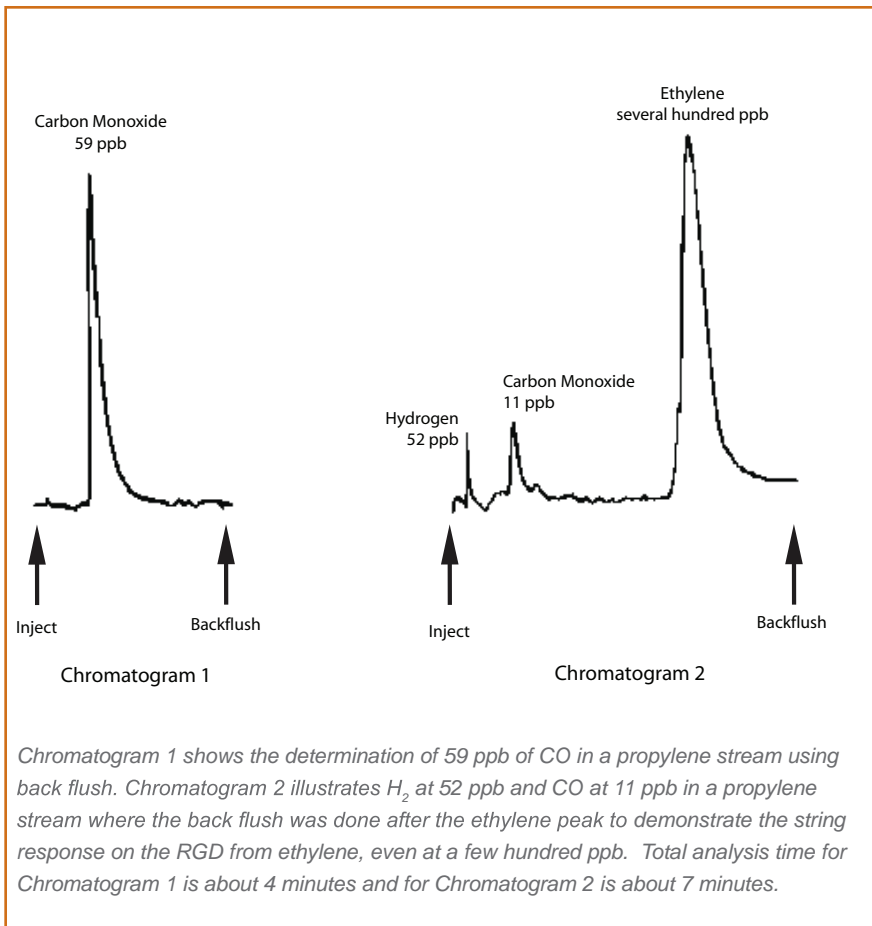

Figure 1. Flow diagram for method P-001.

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Applicaiton Note

Carbon Monoxide
59 ppb

Ethylene
several hundred ppb

Carbon Monoxide
11 ppb

Hydrogen
52 ppb

Inject   Backflush

Chromatogram 1

Inject   Backflush

Chromatogram 2

Chromatogram 1 shows the determination of 59 ppb of CO in a propylene stream using back flush. Chromatogram 2 illustrates H$_2$ at 52 ppb and CO at 11 ppb in a propylene stream where the back flush was done after the ethylene peak to demonstrate the string response on the RGD from ethylene, even at a few hundred ppb. Total analysis time for Chromatogram 1 is about 4 minutes and for Chromatogram 2 is about 7 minutes.

Method P-001 is one of several application packages developed by Trace Analytical. Our applications group is always ready to consult with you about your specific analytical requirements. Please contact AMETEK Process Instruments or your local AMETEK representative for information on our Trace Analytical gas purity monitors.

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