Measurement of Nitric Oxide in Ethylene by Gas Chromatography and Chemiluminescence Detection

Technical Overview

Introduction

Measurement of nitric oxide (NO) in hydrocarbon streams, such as ethylene, is important for helping to prevent potentially hazardous conditions. It is necessary to measure NO in low concentrations, making this a very difficult analysis. Gas chromatography with chemiluminescence detection provides a successful technical solution to this problem.

Presence of NO_x comprised primarily of nitric oxide (NO) and nitrogen dioxide (NO_2), in some hydrocarbon streams can lead to the formation of a potentially hazardous condition. Explosive nitrated resins may form in cryogenic units (cold boxes) under some conditions. In the production of ethylene, NO is of greatest concern because it is the only NO_x species that reaches the cryogenic ethylene recovery unit.

It is desirable to measure NO to as low a level as possible, for example as low as 10 ppb (parts per billion). Trace level analysis of NO is difficult because of the presence of interferences, including both the matrix and trace level contaminants. Many analytical techniques fail in this application, due to lack of the required sensitivity or specificity.

Agilent Technologies has developed a successful approach to this challenging measurement. This approach uses gas chromatography with chemiluminescence detection with the Agilent 255 Nitrogen Chemiluminescence Detector (NCD). A 10-port valve and two-column approach (backflush of analytical column to vent) is used to minimize interferences and analysis time. A simple schematic of this approach is shown in Figure 1.

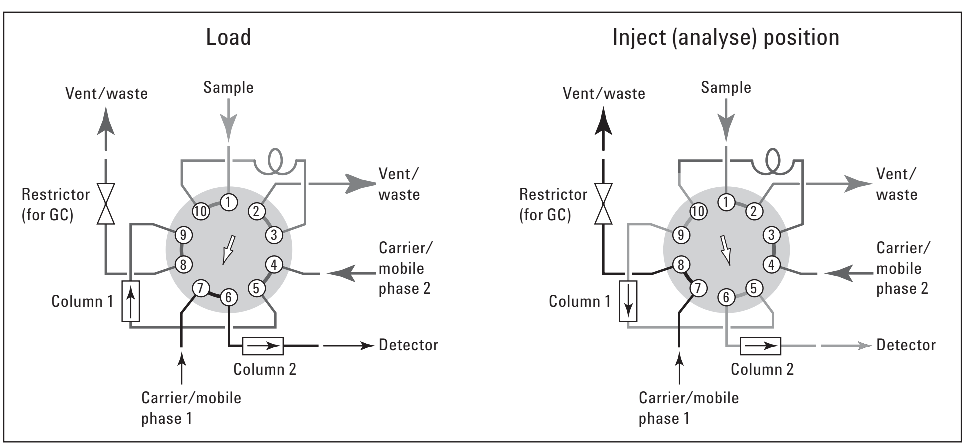

Figure 1. Valve configuration for determination of NO, more retained components are backflushed to vent.

The valve is initially in the load position to fill the sample loop. It is then switched to inject the sample and deliver NO to the detector. The valve is then actuated to backflush the more retained components out the vent, while simultaneously refilling the loop with sample to get ready for the next analysis.

The choice of the gas chromatography columns is extremely important. First, the analytical column (column 1) must separate NO from ethylene. The columns, valve, loop, and connective tubing must not significantly adsorb NO at low levels. This is a difficult challenge, but adsorption of NO can be minimized by the proper selection of materials.

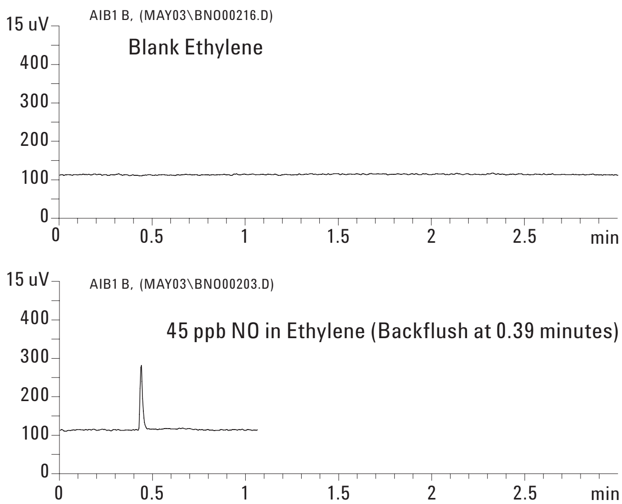

Figure 2. Demonstration of lack of ethylene matrix interference (top) and sensitivity to NO at a low concentration level.

In conclusion, the measurement of NO in hydrocarbon streams like ethylene is an important but difficult measurement. Gas chromatography with chemiluminescence detection provides a successful technical approach to this measurement need. This approach can provide the needed selectivity to minimize potential interferences, as well as the sensitivity required to measure NO at low ppb concentration levels.

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