Trace Impurities in Nitrogen
by Advanced Industrial Chemistry Corporation

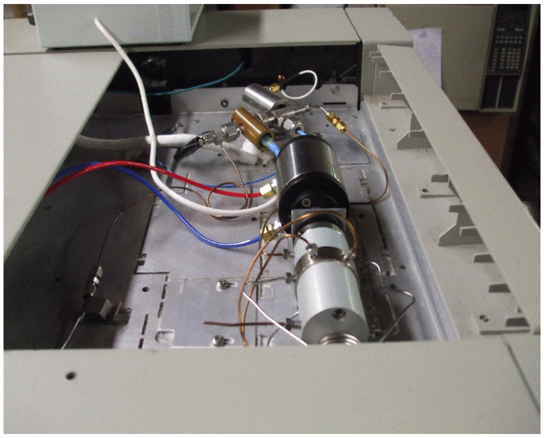

Figure 1:Configuration of Trace Impurities GC.

Air separation units (A.S.U.'s) produce oxygen, nitrogen and argon from atmospheric air using cryogenic distillation. Depending on how the plant is configured, they can produce nitrogen with a higher or lower concentration of argon.

A customer requested a custom configured instrument capable of measuring less than 0.3 ppm hydrogen and less than 30 ppm of argon in bulk nitrogen. Because this customer has real-time on-line instrumentation for oxygen and the oxygen concentration is sub-ppm, a simple GC can be configured to analyze for these two constituents.

The GC configuration

An Hewlett Packard (H.P.) 5890 Series II gas chromatograph (G.C.) was configured with an air actuated six port sampling valve and a two mL sampling loop. This was combined with an A.I.C. dielectric barrier discharge (DBD) helium ionization detector (HID). A picture of this configuration is shown in Figure 1 above. The six port sampling valve was built with a purged head which uses the exhaust of the D.B.D. as the purge gas source to save on the consumption of helium.

The G.C. system was configured with a molecular sieve column without backflush. This configuration is possible since the product stream contains very little moisture or CO_2 as these constituents are diligently removed from the product gases and monitored with on-line analytical systems. The instrument was configured with a standard H.P. manual packed port pneumatics although the carrier gas was plumbed directly to the six port valve without the packed port injector.

The system uses standard copper tubing and plumbing up to and inside of the G.C. without any significant adverse effects upon the chromatography or the detector. This ability to use copper tubing meant that plumbing the system into the facility was a simple matter that took advantage of already existing lines.

Reaction gas to the D.B.D. H.I.D. was helium. The regulated flow for this gas was provided by the make-up fitting from a standard H.P. flame ionization detector (F.I.D.) flow gas control manifold. The D.B.D. H.I.D. uses the standard F.I.D. electrometer card except that the standard signal bar is replaced with a signal cable.

Why choose a 5890 G.C.?

Using a 5890 G.C. chassis, A.I.C. built a G.C. system capable of meeting this customers requirements for about 1/3 the price of a new GC system. The H.P. 5890 G.C. is one of the most widely used G.C.'s in the world. It is an excellent, economical, alternative to buying a brand new G.C. The robust nature of the chassis, the extensive configuration options available and the ease of customization all combine to give customers the results they need at a price they can afford. This application (isothermal analysis, packed column flow control, simple configuration) is an ideal application for the utilization of a 5890 G.C. chassis.

Spare parts for this G.C. system are often a concern for people purchasing this system and there are a number of answers to this concern. First, A.I.C. offers a wide range of parts, from inventory, for the G.C.. Second, Agilent still offers many of the parts for the 5890. Finally, there is a robust market for parts from a number of third party vendors in the United States. Names of such vendors can be provided upon request.

Results

Shown below are two chromatograms generated by the custom G.C. system built by A.I.C. Figure 2 is a full scale view of an analysis of C grade nitrogen from the A.S.U. unit. Two things are most apparent from this chromatogram. First, the argon peak at 3.1 minutes which, in this chromatogram, is running at about 28 p.p.m. The second obvious feature of the chromatogram is the dip in the chromatogram upon the elution of the bulk nitrogen. This dip is due to a large amount of nitrogen (2 c.c.'s) injected which quenches all ionization within the detector for a brief period of time. This quench has no lasting effect on the operation of the detector and, in fact, the detector recovers in a matter of minutes after the elution of the nitrogen peak.

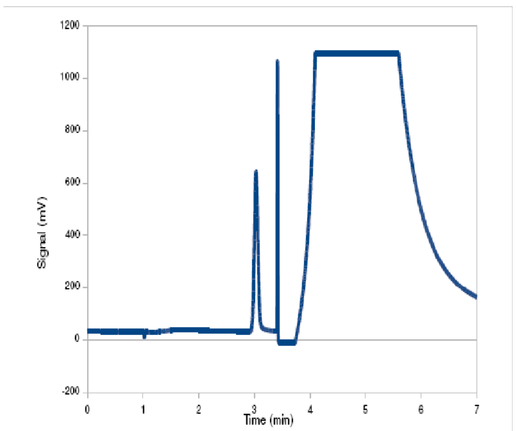

Figure 2: Chromatogram from C grade N2 analysis

Figure 3 below is a zoomed in view of the section of the chromatogram that contains the hydrogen peak for the standard used in this analysis. This standard mixture has 0.9 ppm of hydrogen in it which is clearly evident in this chromatogram with excellent signal to noise. Note also the valve fluctuations in the chromatogram at 1 minute and 1.6 minutes, relatively minor considering the use of a 2 c.c. loop. This is another of the performance characteristics of the D.B.D. H.I.D., it's excellent behavior in response to valve actuation.

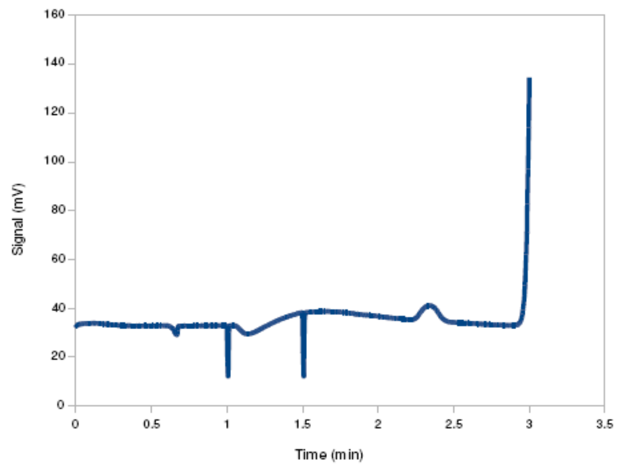

Figure 3: Close up view of the hydrogen peak in the standard chromatogram

Advanced Industrial Chemistry

Advanced Industrial Chemistry (A.I.C.) specializes in unique gas chromatographic detectors and in building custom gas chromatographic systems around those detectors. The ability to match the detector to the application means that A.I.C. can build systems that will specifically meet customer applications with out a lot of bells and whistles.

A.I.C. has developed methods and built custom G.C. systems for a number of industries including environmental, petroleum, consumer products, and research and development. With over 13 years in the gas chromatography business, A.I.C. offers the experience and the know how to meet customer needs in a cost effective manner.

Other Applications

Examples of other custom built instruments available from A.I.C. include:

Measurement of low p.p.m. levels oxygenates in L.P.G.

Measurement of syngas fixed gases, including H2.

Measurement of p.p.m. carbonyl sulfide in CO2 using A.I.D.

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