"BEYOND THE ORDINARY": SAMPLE SYSTEM BEST PRACTICES FOR CRYOGENIC MOISTURE AND HYDROCARBON DEW POINT APPLICATIONS

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Abstract

Sampling techniques for BTU and moisture measurements are well understood but applications beyond the routine requires additional attention to detail. Two such applications are the subject of this paper. The first is the measurement of low level moisture (1 – 1,000 ppbv) for cryogenic processes. Measuring moisture below 1 ppm is an entirely different world from the traditional pipeline application because of the highly polar nature of the water molecule, as it tenaciously adheres to all surfaces in the sample system. Without consideration of these surface effects, sample transport and subsequent analyzer response is compromised.

The second is measurement of the hydrocarbon dew point using a continuous device, a physical property rather than a quantitative method. The challenge is to sample the gas at pressure (or alternatively at a fixed inlet pressure), while separating oil, grease and liquid hydrocarbon without affecting the integrity of the sample gas.

Introduction

Mole sieve dryers at the inlet to the cryogenic recovery plant must dry the feed gas to a moisture concentration of typically less than 100 parts per billion (ppb, or 0.01 ppm). This concentration of moisture is only 1/1000th of the moisture limit that is commonly tolerated in pipeline quality natural gas. Too much moisture will lead to a loss of recovery efficiency and, if undetected, damage to the process equipment. Moisture dew points, needed for process control at these temperatures, test the ability of the analyzers to measure them. A well thought out, designed and executed sample system is essential if successful analyses are to be made.

Automated hydrocarbon dew point detection for applications where natural gas is processed, transported, stored or used as a feedstock (for natural gas fired gas turbines) is critical to determining the quality and the characteristics of the gas stream to be monitored. Sampling systems must be designed to ensure that the stream to be measured is representative of the process conditions. A system should be designed to ensure that it is suitable for the analysis method (technology) tasked to perform the measurement, while at the same time ensuring that none of the key constituents in the sample are removed or compromised.

The Sample System

As has been mentioned, there are six overall functions of any sampling system.[1]

- To take a sample that is representative of the flowing process stream, relative to the application needs of the particular system.
- To transport the sample from the sample point to the analyzer.
- To condition the sample so it is compatible with the analyzer and the application needs. Conditioning may include cleaning, vaporizing, condensing, adjustment of pressure and temperature.
- To switch from one stream to another, where the analyzer is used on more than one sample stream. This may include the introduction of calibration standards.
- To transport the sample from the analyzer to the point of disposal.
- To allow for the effects of corrosion and other reactions.

[1] E.A. Houser, *Principles of Sample Handling and Sampling Systems Design for Process Analysis* (Pittsburgh: Instrument Society of America, 1972), 3

Essentials of a Cryogenic Sample System

The measurement of ppb moisture in cryogenic process samples (turbo expansion) is one of the most difficult measurements made. In the world of semiconductor gas measurement, ppt moisture levels are common. There, it is said the analyzer and sample system cannot be separated. They are both essential to the analysis. This is also true of natural gas cryogenic process measurements. This point cannot be stressed enough. Analyzer manufacturers who specialize in providing process analyzers for these applications generally understand the key analyzer characteristics required to ensure that the sample is suitable for the analyzer technology as well as the application for which it is designed.

The water molecule is sticky. It sorbs (forms an equilibrium layer) onto all known surfaces. This effect slows response time to moisture level changes. It has been demonstrated that electro polished 316L (semiconductor grade) tubing must be heated to 350^{o}C before there is enough thermal energy present to prevent the water molecule from sticking (Martinez, J, NIST). While it may be possible to achieve this in a laboratory, it is generally not possible or practical to heat tubing or sampling system components to these temperatures in the field.

Although it may not be possible to completely eliminate the sorption effect the inner surface of a sample system has on a cryogenic process measurement, all one can do is work diligently to minimize the impact. Sample system fundamentals, important to ensuring that the analyzers tasked to perform these critical measurements operate properly and are provided a representative sample, are often taken for granted. The ideal, most practical, but not often realized key points are described as follows:

- Extracting the sample from the process - The **probe** is the door to the process. The measurement starts here. There are many different probe designs and manufacturers who specialize in probes for natural gas applications, including the critical application of measurement of low concentrations of moisture in cryogenic applications and hydrocarbon dew point analysis. The most common and most acceptable types of probes include (but are not limited to) simple designs, which consist of a piece of pipe inserted into the process. Probes may be designed to be inserted and removed under pressure. Some probe designs serve as in-the-pipe pressure reducers, others have membrane filters to trap liquids for return to the process. The probe's inside diameter should be minimized and the inner surface should be electro polished and passivated. When possible, the probe should be inserted into the top of the process pipe and into a depth of about one third of the way across. This ensures that the analyzer system 'grabs' a representative sample from the process. Never sample off the wall of a pipe. All sorts of contaminants lurk there, which typically consist of liquids which travel along the walls of the pipe, or solid particles (such as corroded metal from the pipe). These contaminants can cause an analytical system to break down before a sample has reached the analyzers. A probe will avoid these issues and should not be left out.
- The first step of sample conditioning - if **pressure reduction** is needed, it should be performed at the probe, or as close to the process connection (the probe location) as possible. Besides reducing sample pressure to an acceptable level, pressure reduction at the probe results in increased volumetric flow which improves speed of response. The pressure reducer must be in the pipe, or heated to prevent problems with Joule-Thompson cooling. In-the-pipe pressure reduction works well, but heated pressure reducers installed at the probe are typically acceptable. Heated pressure reducers should be installed as per vendor requirements, and careful material selection (304 or 316 SS as a minimum) should be considered.
- Transporting the sample to the analyzer - the **sample line** is used to transport the sample from the probe (or pressure reduction stage) to the analyzer. A sample line should be kept as short as possible. Minimizing sample line length limits the opportunity of the moisture molecule to 'stick' to excess surfaces; less surface, less opportunity for losses. Sample line material for applications in cryogenic sample systems should be of 316L, passivated and electro-polished stainless steel. A key requirement to ensuring that a representative sample is transported through the lines is to keep the sample line clean. Sample line temperatures should be kept hot. A general recommendation is to keep the sample line temperature as hot as possible and practical, consistent with process considerations and safety (hazardous area requirements). There is generally no 'magic' temperature (other than 350^{o}C), however, general requirements are to keep the sample line temperature well above ambient temperatures. The temperature set point should be kept constant, maintaining a constant temperature is as important as keeping it hot. Insulated, electrically controlled heated bundles are generally well accepted and meet these requirements. The inside diameter of the sample line should be kept as small as possible

consistent with sample flow requirements (no larger than ¼ inch OD for cryogenic sampling). All of these recommendations and general guidelines help minimize the sample lines inner surface stickiness and surface area.

- Additional components of a sample system - **Fittings** should be tube-type and kept to an absolute minimum in number. Elbow fittings, connectors, bulkhead fittings should be avoided or minimized. It should be noted that all non-welded fittings eventually leak, this is unavoidable. Fittings, even when they do not leak provide virtual leaks, places where the water molecule can hide to be released into the sample line or downstream sample system later. The general rule to follow when designing sample systems for cryogenic moisture analysis is to keep any additional fittings and components to a minimum. This includes avoiding potential dead-legs (pressure gauges are dead-legs, keep these to a minimum) in the system. If pressure sensing or flow monitoring equipment is required, it is generally recommended to source transducers and flow meters suitable for very pure (HP/UHP) gases. These devices are designed and built with minimum surface areas and can be obtained in electro-polished and passivated configurations.

As is the case with every sample system, a system designed for low-level detection of moisture in cryogenic applications should be kept simple. A simple design ensures minimal maintenance requirement and reduces/eliminates surface areas.

Sample Systems for Automated Hydrocarbon Dew Point Analyzers

Many of the recommendations described for sampling systems designed for monitoring moisture in cryogenic applications can be applied to the monitoring of hydrocarbon dew point temperatures in natural gas and natural gas related applications. There are a few differences related to sample systems designed for automated hydrocarbon dew point analyzers which will be described and discussed.

Automated hydrocarbon dew point analyzer technology is generally very similar to the traditional manual method, consisting of a chilled mirror device that cools a mirrored surface in contact with the flowing gas to the temperature at which the hydrocarbon condenses on the surface of the mirror. Most automated dew point detection technology requires a cyclic measurement system, whereby the sample is collected, trapped in the sample system, cooled to the temperature at which the dew point forms, warmed and then purged to allow a fresh sample to enter the analyzer sample cell after which a new sample measuring (or cooling cycle) begins. Sample cycle times vary depending on the analyzer vendor.

Hydrocarbon dew point determination may be performed at line pressure or at fixed pressures, depending upon the requirements of the user and how hydrocarbon dew point temperature is to be reported. When a fixed pressure measurement is required, pressure reduction should consist of either an in-situ pressure regulating sample probe or an external, heated pressure reducing regulator as described previously.

- Monitoring dew points at **line pressure** – a sample system designed to monitor hydrocarbon dew points at process pressure does not require pressure reduction. A sample probe, as described in the cryogenic moisture analyzer application is required to ensure that a representative sample is provided to the analyzer. Components in the sample system should be kept to a minimum to ensure the least amount of pressure drops across the sample system. Any system components which trap the sample should be installed downstream of the sample cell to allow the sample detection cell to reach equilibrium with the sample pressure. Recommendations with regards to sample lines, sample line lengths, materials of sample lines and sample system components as described for the cryogenic moisture application should be followed for the hydrocarbon dew point analyzer application.
- **Sample filtration** – monitoring for dew points in pipeline specification natural gas applications requires that the sample be properly treated for potential contaminants in the stream. Sample systems should incorporate filters based on membrane technology to ensure that contaminants be prevented from entering the sample cell or critical parts of the sample system of the analyzer. A sample system should incorporate the means of also preventing particulate matter from entering a sample cell or a sample system. Coalescing filters have been tried and tested in this application. Preventing contaminants from entering a measuring cell or being deposited on the surface of a chilled mirror will enhance the performance of the analyzer and reduce maintenance intervals required to maintain the system.
- **Stream switching** – although not generally recommended, there may be instances or situations where a stream switching system may be possible or economically feasible for determining hydrocarbon dew point

temperatures in natural gas applications of similar gas compositions. Sample system components such as solenoid valves (for switching between streams) should be designed for high pressure applications. In addition, it should also be noted that, since automated chilled mirrors are cyclic measurement devices, ample time be allocated between cycles. Generally, several measurement cycles may be required for the analyzer to stabilize between switching streams.

- **Ambient temperature control** – automated chilled mirror devices benefit from a controlled ambient temperature environment. If external ambient temperatures change drastically, it is generally recommended to install the analyzer and sample system at a controlled ambient temperature.
- **Calibration, verification** – chilled mirror devices used for the detection of hydrocarbon dew point temperatures of natural gas generally follow a first-principle methodology. However, sample systems should be designed to have the ability to 'simulate' a hydrocarbon dew point temperature of a hydrocarbon. A verification system, to include the ability to introduce a hydrocarbon at a fixed pressure can help trouble-shoot potential analyzer problems or provide a means to periodically check the analyzer and sample system to ensure proper operation. Care should be taken to follow vendor recommendations on set up of the verification system.

Conclusion

Choosing the appropriate technology for measuring critical components in natural gas processing applications is important to ensure that accurate information is provided to the operator of the process. However, care should be taken to ensure that the sample system is designed to provide the analyzers with a representative sample of the process. A carefully designed sample system ensures high reliability, low maintenance intervals and meets overall performance objectives. Following some simple yet key guidelines will ensure a successful analyzer system installation. A key concept to follow in sample system design for these critical natural gas analyzer applications is to 'keep it simple', reduce/remove excess components, and choosing materials suitable for the service.