Introduction

Industrial gas manufacturers package and deliver gases in a variety of different ways. These include on-site pipeline distribution, bulk delivery of liquefied gas into an on-site bulk container, exchange programs for large compressed gas vessels (i.e., tube trailers), and delivery of smaller cylinders containing gas or liquefied gas. Each delivery mechanism has unique advantages, but also unique associated costs. Here we describe the requirements and challenges of tube trailer filling, detailing the needs related to filling high purity Helium into tube trailers and showing that Tiger Optics’ Spark H₂O analyzer can offer a perfect solution for detecting trace moisture in this application.

Challenges of Helium Filling

Helium tube trailer filling is a demanding activity with relatively high cost and process complexity. “Crude” Helium is harvested in geographic locations that have Helium-rich natural gas fields, which mostly include areas in Algeria, Australia, Canada, Poland, Qatar, Russia, and the United States (see Figure 1). It is delivered to industrial gas packaging facilities around the world as a bulk cryogenic liquid in 11,000-gallon refrigerated ISO tankers. The bulk liquid is not thermodynamically stable and must be quickly and efficiently transferred to more stable product forms (i.e., pressurized gas) with longer storage potential (see Figure 2). This is typically accomplished by decanting off some of the liquid into smaller dewar vessels for immediate sale and use, and gasifying and compressing most of the Helium for filling pressurized vessels (compressed gas cylinders and “tubes”). In recent years, Helium has become short in supply numerous times. As a result, Helium prices have doubled in the last decade and are routinely exceeding $200 per thousand standard cubic feet (SCF). Therefore, efficient management of Helium processing and minimization of product loss directly affects the profitability of the enterprise. Additionally, Helium tube trailer filling is a time-consuming process, taking at least 14 hours for a successful fill of the largest tube bundles (180,000 SCF). With respect to other activities at packaged gas facilities, a relatively large investment in time and product value is made for Helium tube trailer products.

11,000 gallons liquid He

Decant liquid

Pressurize and fill cylinders

20,000 to 180,000 standard cubic feet

Pressurize and fill tube trailer

Fig. 2 Typical packaging of bulk liquid Helium for storage and delivery

Fig. 1 Worldwide Helium production per country (in percent)

Worldwide Helium Production per Country (2016)

56%

31%

6%

3%

1%

<1%

2%

<1%

United States
Algeria
Australia
Canada
Poland
Qatar
Russia
Others

Typical Helium tube trailer filling operations perform several steps including gasification, compression, purification, and tube filling (schematically outlined in Figure 3). The goal is to fill the trailer tubes in the most energy and time-efficient manner possible, and there are several points where making a fast, accurate moisture measurement can assist in achieving this goal. The major monitoring points of interest are:

- **Incoming Helium Product**: Incoming crude Helium is typically too wet to be packaged without some purification. Moisture measurements made on the crude Helium allow optimization of purification procedures.

- **Post-Purification**: Purification processes typically include a selective adsorptive bed that can saturate with moisture and other impurities. Once saturated, the bed loses its capacity to retain any additional moisture and it must be regenerated. The active lifetime of the bed is a function of the moisture level in the incoming gas stream and the total time of usage. Monitoring the purification system output stream helps to indicate the active lifetime of the bed between regenerations.

- **Tube Trailer Inlet**: Most crucially, monitoring of the Helium product as it enters the tubes assures that the tube trailer is filled with in-specification product. Moisture measurements made at the point of fill will detect any quality defect stemming from bad product, inadequate purification, or compromised transfer lines. Since the fill is accomplished over such a long period of time, small variations in the moisture levels can indicate to an operator that minor process adjustments should be made. For example, an upward-shifting moisture level could indicate that the purification must be slowed, to better extract the moisture.

Traditional moisture measurement technologies have some drawbacks for these applications. Most demonstrate slow response time, limited range, and a high degree of signal averaging, which can miss a moisture excursion. At minimum this can lead to energy waste in the process, and in the worst case an entire tube trailer can be filled with out-of-specification product due to a missed moisture spike. Lack of ability to change sampling points is another issue with traditional technologies. To switch from crude product to purification or final fill sampling points, the analyzer needs to be disconnected and the inlet exposed to ambient for a short period. Most technologies would experience saturation in this situation and take hours or even days to dry down again sufficiently to perform the next measurement.

**Improving Measurement with the Spark**

For each of the measuring points highlighted above, Tiger Optics’ Spark H₂O analyzer (see Figure 4) is optimally suited to make the measurement. The absolute accuracy offered by Cavity Ring-Down Spectroscopy (CRDS) is a key advantage of Tiger instrumentation. There is also no requirement for a zero gas or a span gas. The CRDS technique offers an intrinsic zero
at a spectral point of no absorbance, and the Spark contains an internal laser-lock reference cell that assures quantitative accuracy.

With the Spark, the time formerly spent on zero and span calibrations with legacy technologies is time used making measurements. And the analyst can have the utmost confidence that they are getting the most accurate measurement possible.

In addition to the outstanding accuracy, the Spark $\text{H}_2\text{O}$ offers the user industry-leading response speed, due to its fast, non-contact spectroscopic technique and its low cell volume and low amount of wetted surface area. In fact, the Spark can dry down to sub-ppm readings in under 3 minutes in its initial installation. This characteristic gives the user the flexibility to sample several different measurement points in a short time period, without needing a complex, engineered sample switching system. Pairing this response speed with the small, lightweight design of the Spark, the user can move the instrument from sample point to sample point with ease, speed and confidence. Figure 5 offers a representative look at the Spark’s typical fast response speed and excellent repeatability to a 11 ppm moisture intrusion.

Cavity Ring-Down Spectroscopy

All Tiger Optics instruments are based on CRDS. The key components of the CRDS system are shown in Figure 6.

CRDS works by tuning laser light to a unique molecular fingerprint of the sample species. By measuring the time it takes the light to decay or "ring-down", you receive an accurate molecular count in milliseconds. The time of light decay, in essence, provides an exact, non-invasive, and rapid means to detect contaminants.

Tiger Optics Overview

Tiger Optics introduced the world’s first commercial Cavity Ring-Down Spectroscopy (CRDS) analyzer in 2001. Today, our instruments monitor thousands of critical points for industrial and scientific applications. They also serve the world’s national metrology institutes, where they function as transfer standards for the qualification of calibration and zero gases.

First ISO-Certified CRDS Company

Tiger Optics is the first CRDS company certified to the ISO 9001:2008 and the current ISO 9001:2015 standard of process consistency and continuous quality improvement.

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