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Spark • HALO 3 • HALO H2

Cost-Effective Purity Analysis in the Cryogenic Air Separation Process

This application note details the use of Process Insights' Spark and HALO series of Cavity Ring-Down Spectroscopy (CRDS) analyzers for detection and analysis of moisture, carbon dioxide, methane, acetylene and hydrogen in the cryogenic air separation process.

Improve Safety & Process Efficiency with CRDS

Process Insights' trace gas analyzers offer many opportunities to improve the air separation process by saving time & money, and alerting plant operators quickly in case of unsafe impurity levels.

Key advantages of CRDS analyzers include:

- Freedom from calibration
- No consumables or service gases required
- All solid-state design, no moving parts
- Plug-and-play, easy to operate
- Accurate detection of H_2O, CO_2, CH_4, C_2H_2 and H_2
- Fast speed of response, ideal for process control

Cryogenic Air Separation

Cryogenic air separation units (ASUs) are the gas industry's workhorses for the production of gaseous and liquid highpurity nitrogen, oxygen and argon. The cryogenic process can be modified to manufacture a range of desired products and mixes. Even so, it typically calls for sequential steps, which are outlined in Figure 1.

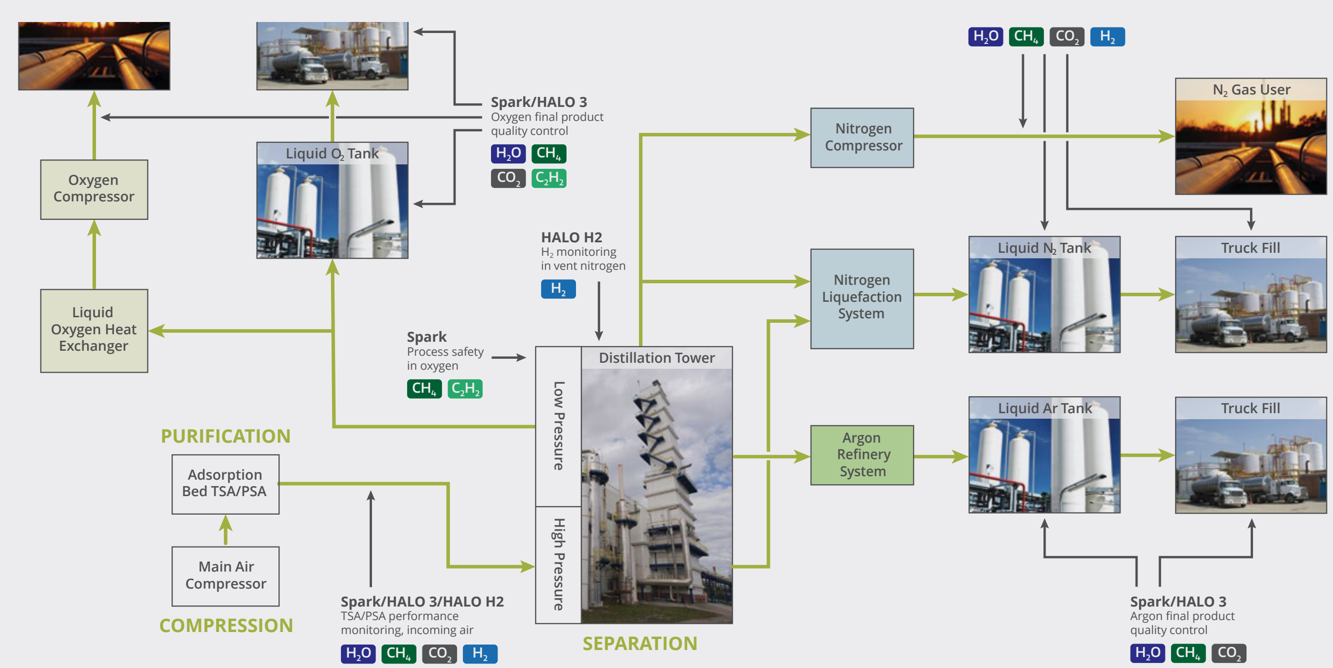

Fig. 1 Cryogenic air separation steps and monitoring points

Controlling Impurities to Ensure Safe ASU Operation

Following compression, the air pre-treatment step consists of cooling and purification to remove process contaminants, such as H_2O, CO_2 and others. The most common purification methods are Temperature Swing Adsorption (TSA), which exploits the difference in adsorption capacity of adsorbents at different temperatures, and Pressure Swing Adsorption (PSA), which operates similarly via pressure variations.

Purification is essential to this process, as impurities can freeze in the downstream heat exchangers and cryogenic separation equipment. As a result, product quality may be impaired, and the system itself can suffer damage. H_2O and CO_2, for instance, solidify at cryogenic temperatures and cause plugging of lines and valves, leading to plant malfunction and possible damage.

Hydrocarbons condensing in the product cause a different issue, which specifically affects oxygen. Because molecules like CH_4 or C_2H_2 have a boiling point higher than oxygen, impurities can concentrate in the liquid product. Excessive amounts of these volatile hydrocarbons in pure oxygen can cause rapid and violent oxidation, which poses an enormous safety risk. While piping for oxygen service is generally cleaned to the highest standards, any small particle released from a surface and moving at high speed can become a potential ignition source. Therefore, monitoring certain hydrocarbon impurity contents is essential to maintain safe levels.

Hydrogen is another contaminant whose monitoring is important to maintain control over the air separation process. Since H_2 cannot be removed during the process, nitrogen venting is a way to eliminate excess H_2; therefore, monitoring the H_2 levels in the vent N_2 is an important indicator of the capability of the process to control the H_2 content in the gas.

How Process Insights can help

To avoid damage to the plant and harm to workers, our CRDS analyzers can efficiently monitors the air emitted from the TSA and PSA to ensure complete H_2O removal prior to entering the heat exchangers, and CO_2, CH_4, C_2H_2 and H_2 levels within safe operating limits.

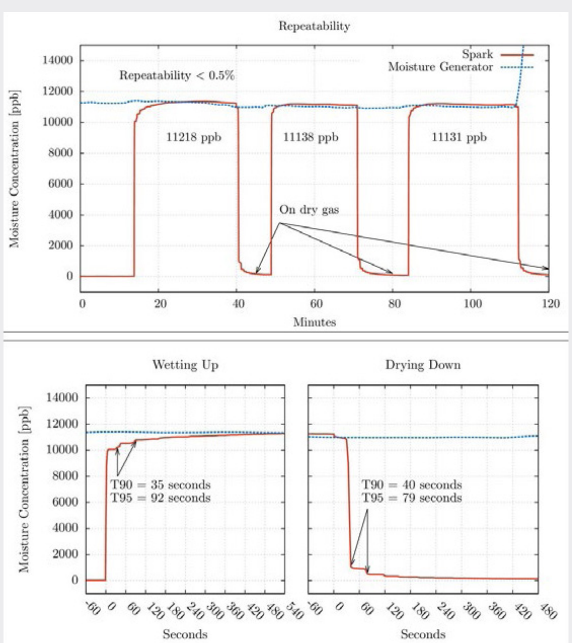

Fig. 2 Repeatability and speed of response of the Spark H_2O

In general, ASU operators can choose between the costefficient Spark series and highly sensitive HALO series to monitor impurities. The choice depends largely on the purity requirements of the end product. For moisture analysis, for example, the Spark measures H_2O in N_2 down to 12 ppb over a wide concentration range from 0 to 2000 ppm, whereas the HALO 3 measures as low as 1.2 ppb with a 20 ppm upper range. As shown in Figure 2, the Spark's accuracy is within 15 ppb and the repeatability is better than 0.5% (RSD) for repeated intrusions. With a specified speed of response of less than 3 minutes to 95% of intrusion level, the Spark provides fast moisture detection. As the results show, the response is often faster than our stated specification in practice, when operated with an optimized sampling system.

The Spark CO_2/HALO 3 CO_2, Spark CH_4/HALO 3 CH_4 and Spark C_2H_2 are ideal replacements for non dispersive infrared (NDIR), flame ionization detectors (FIDs) or gas chromatographs (GCs). With their fast speed of response to detect excursions, our CRDS instruments are best for process control. Unlike NDIR, CRDS users achieve freedom from drift, which means no cumbersome calibration and maintenance protocols. In addition, using CRDS for CH_4 and C_2H_2 is a safer alternative FID, as no hydrogen fuel gas is required. Users also eliminate all of the calibration and utility gases that go along with the FID or GC. This typically saves up to eight cylinders! CRDS also provides real-time 24/7 measurements as well as excellent sensitivity (LDL of 6 ppb for the Spark CH_4 in O2, 1.1 ppb for the HALO 3 CH_4 in O_2, 7 ppb for the Spark C_2H_2 in O_2 and 8 ppb for C_2H_2 in N_2) to ensure that

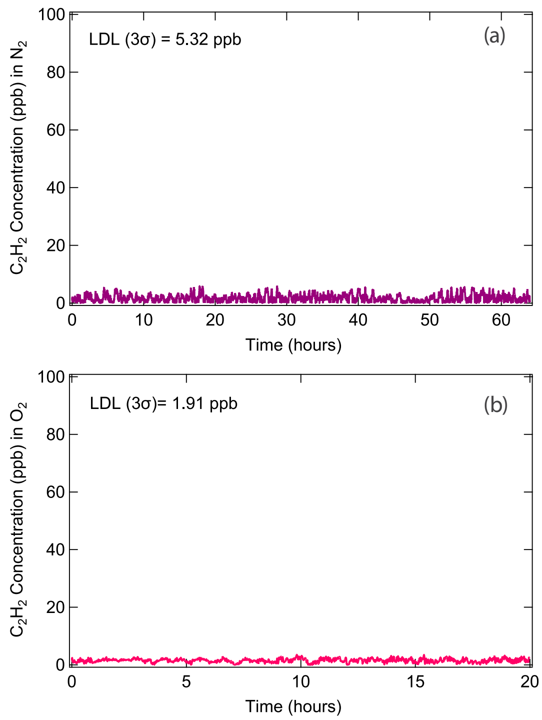

Fig. 3 Repeatability and speed of response of the Spark H_2O

excessive impurity levels are immediately detected to ensure safe operation of the plant at all times. Figure 3(a) shows a more than 60-hour-long measurement with the Spark C_2H_2 in zero N2 well below the specified LDL. Similarly, Figure 3(b) shows baseline readings in O_2 over 20 hours well below LDL. The Spark C_2H_2 also excels during span testing. The measurement in N_2 uses a certified reference mixture (3961 ppb C_2H_2), span testing in O2 is performed with a 200-ppb sample generated by a dilution system. As shown in Figure 4(a), relative accuracy of -0.05% and precision of 0.19% are well within the guaranteed specifications and underscore the Spark's stellar performance. The oxygen data also shows excellent relative precision with 0.54%.

In addition to its low detection limit and high precision, the Spark C_2H_2 also excels in speed of response. Figure 5 demonstrates zero-to-span and span-to-zero response times well below one minute (with span level of 200 ppb). Figure 6 shows similar data for the Spark CH_4 at a higher span concentration of 2 ppm. These results underscore the Spark's ideal application as a continuous, real-time sensor for safety critical measurements.

To monitor trace hydrogen, Process Insights offers the HALO H_2. Because H_2 cannot be detected directly with near-IR light, the analyzer is based on Tiger's proprietary catalytic conversion process, most notably used in Tiger's trace oxygen analyzers and field-proven for more than a decade. The conversion of H_2 to H_2O allows sensitive detection with CRDS while offering 100% conversion efficiency for reliable calibration. Figure 7 shows the HALO H_2's zero baseline in N_2 for a period of almost 150 hours (more than six days). No drift or spiking is observed and the 3σ-noise is only 4.10 ppb and therefore well below the guaranteed LDL of 8 ppb.

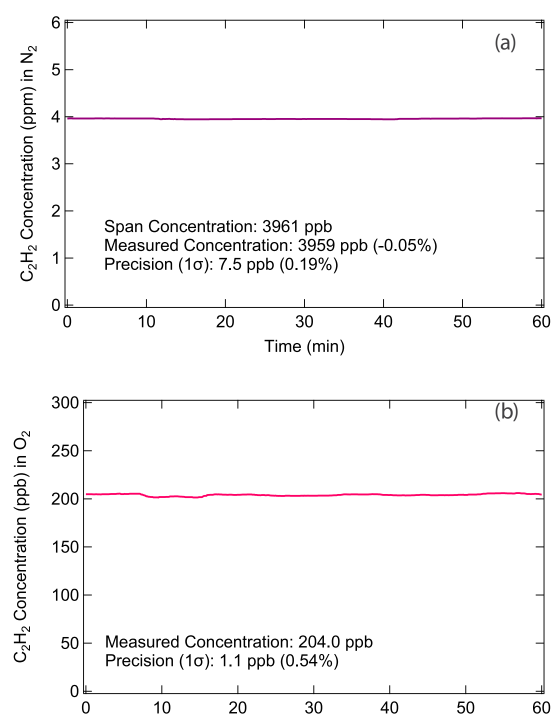

Fig. 4 Spark C_2H_2 span measurement in (a) nitrogen and (b) oxygen

Table 1 shows a summary of all detection specifications of Process Insignts' Spark and HALO analyzers for typical gases used in the ASU process.

Table. 1 Summary of Spark and HALO detection specifications

Analyzer	Gas Matrix	Range	LDL (3σ/24h)
Spark H_2O	Air (CDA)	0 – 1800 ppm	10 ppb
	Ar	0 – 900 ppm	4.5 ppb
	N_2	0 – 2000 ppm	12 ppb
	O_2	0 – 1000 ppm	6 ppb
Spark CO_2	Air (CDA)	0 – 1500 ppm	250 ppb
	Ar	0 – 1200 ppm	220 ppb
	N_2	0 – 1500 ppm	250 ppb
	O_2	0 – 1200 ppm	220 ppb
Spark CH_4	Air (CDA)	0 – 80 ppm	7.5 ppb
	Ar	0 – 70 ppm	6.5 ppb
	N_2	0 – 80 ppm	7.5 ppb
	O_2	0 – 50 ppm	6 ppb
Spark C_2H_2	Air (CDA)	0 – 80 ppm	8 ppb
	Ar	0 – 80 ppm	8 ppb
	O_2	0 – 70 ppm	7 ppb

Analyzer	Gas Matrix	Range	LDL (3σ/24h)
Halo 3 H_2O	Air (CDA)	0 – 18 ppm	1.2 ppb
	Ar	0 – 9 ppm	0.6 ppb
	N_2	0 – 20 ppm	1.2 ppb
	O_2	0 – 12 ppm	0.7 ppb
Halo 3 CO_2	Air (CDA)	0 – 10 ppm	8 ppb
	Ar	0 – 10 ppm	8 ppb
	N_2	0 – 12 ppm	8 ppb
	O_2	0 – 10 ppm	8 ppb
Halo 3 CH_4	Air (CDA)	0 – 7 ppm	1.5 ppb
	Ar	0 – 7 ppm	1.4 ppb
	N_2	0 – 8 ppm	1.6 ppb
	O_2	0 – 6 ppm	1.1 ppb
Halo C_2H_2	Air (CDA)	0 – 5000 ppm	80 ppb
	Ar	0 – 200 ppm	6 ppb
	O_2	0 – 500 ppm	8 ppb

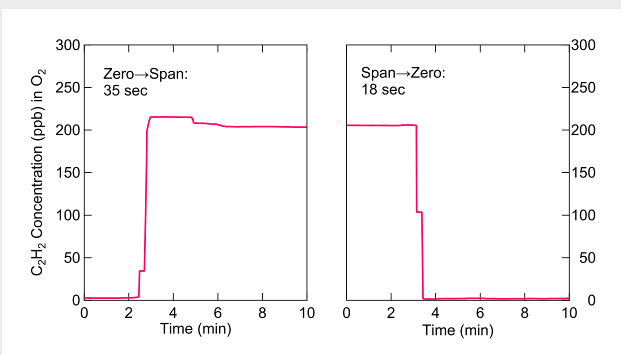

Fig. 5 Speed of response test results for the Spark C_2H_2 in oxygen

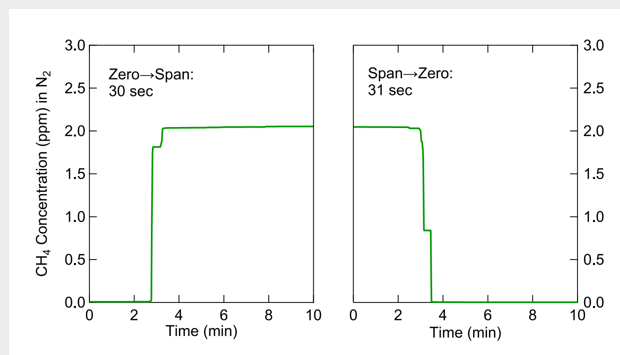

Fig. 6 Speed of response test results for the Spark CH_4 in nitrogen

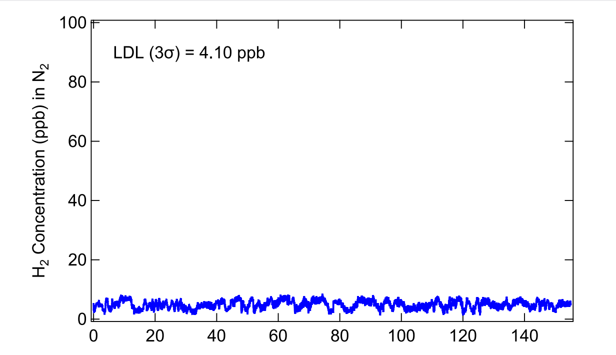

Fig. 7 HALO H_2 zero baseline in nitrogen

Rapid Stream Switching

It is possible to couple any Spark or HALO with a high-purity gas stream selector and eliminate the need for multiple analyzers. Whereas traditional measurement equipment requires dedicated analyzers for the different gases, CRDS's fast speed of response supports rapid cycling between many gas streams for accurate, real-time impurity measurements in all process gases with a single analyzer. Sparks and HALOs also offer seamless integration into the stream switching control system via their standard remote interfaces.

Cavity Ring-Down Spectroscopy

All Process Insights instruments are based on CRDS. The key components of the system are shown in Figure 8. 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-contact and rapid means to measure contaminants.

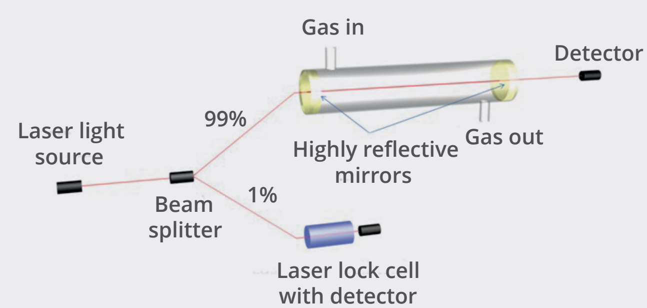

Fig. 8 Principle of Cavity Ring-Down Spectroscopy

Process Insights Overview

Process insights 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

Process insights 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.

GAIN REAL-TIME INSIGHT INTO YOUR PROCESS

Process Insights manufactures and delivers premium sensors, monitors, detectors, analyzers, instrumentation, and software that are mission-critical to keep your operations, personnel, and the environment safe – every day across the globe.

Get the most reliable, precision analytical technologies available on the market today. We will work to match your needs and budget, and provide the optimal, and most stable process analysis solution for your application.

CENTERS OF EXCELLENCE | PROVIDING PROVEN SOLUTIONS

Process Insights is committed to solving our customers' most complex analytical, process, and measurement challenges everyday.

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