Application Note #27

Validating CRDS for Moisture Analysis in Medical Oxygen

Spark™

GASES & CHEMICALS | CEMS | ENERGY | SEMI & HB LED | ATMOSPHERIC | LAB & LIFE SCIENCE

Introduction to Medical Oxygen

Medical oxygen is one of the most commonly used gases in the healthcare industry, from giving O_2 to critical care patients, providing the basis for anesthesia, to supplementing O_2 to patients with chronic lung diseases, such as COPD.

To ensure that the oxygen meets the necessary quality to prevent harm to patients, strict standards outline limits to a variety of possible impurities in the gas, one of them being water vapor (H_2O). One of the most common standards for medical oxygen is the European Pharmacopeia (EP) standard[1], and in this Application Note, we demonstrate analytical equivalency between Tiger Optics' Spark Cavity Ring-Down Spectroscopy (CRDS) analyzers and demonstrate analytical equivalency to traditional electrolytic moisture analyzers, so the Spark can be used as a more modern and powerful alternative.

Proving Equivalency to European Pharmacopeia

The EP standard dictates that the maximum water vapor content in medical and pharmaceutical grade gas must be less than 67 parts per million (ppm), and the recommended method for analysis of moisture content in medical gases is electrolytic-based sensors. Since this standard was published in 1999, gas manufacturers have significantly improved their process efficiency, resulting in considerably higher purity product; at the same time, the state-of-art in analytical technologies for moisture measurement has evolved. The combination of improved analytical capabilities and higher purity product creates an opportunity for gas manufacturers to maximize the return on oxygen by qualifying it for multiple uses in a single validation step.

Based on powerful, proven CRDS, Tiger Optics' Spark H_2O offers a wide dynamic range, from single-digit parts-per-billion to one thousand ppm for analysis of moisture in oxygen. This low-cost, fast and accurate analyzer features self-zeroing and auto-verification, eliminating the need for field calibration and saving time & money on labor and consumables. In addition to qualifying oxygen, the same analyzer can service nitrogen, argon, helium, hydrogen, clean dry air, and many other gases and mixtures.

In support of the proposed use of the Tiger Optics Spark for qualification of medical oxygen, we present the following validation data, demonstrating equivalency in accuracy of the Spark H_2O with two EP-approved electrolytic moisture analyzers. The specifications for these analyzers are summarized in Table 1. In this application note, we highlight several key parameters that were evaluated in the comparison: accuracy & linearity, speed of response, and lowest detection limit (LDL).

Table 1. Analyzer specifications summary for moisture measurement

Specifications	Electrolytic H_2O Analyzers	Tiger Optics Spark H_2O
Measurement Range	0–1000 ppm H_2O in O_2	0–1000 ppm H_2O in O_2
Detection Limit (LDL)	1 ppm	0.006 ppm
Accuracy	± 5% or 0.5 ppm, whichever is greater	± 4% or 0.006 ppm, whichever is greater
Speed of Response	T90 < 20 min (typical) T95 < 30 min (typical)	T90 < 1 min T95 < 3 min
Flow Requirements	100 sccm (measurement is flow-dependent)	<1000 sccm, flexible (measurement is not flow-dependent)
Gas Matrix Compatibility	Cell-specific for O_2 and H_2	N_2, Ar, O_2, H_2, He, CDA
Maintenance Requirements	Cell replacement & calibration every 12–18 months	None

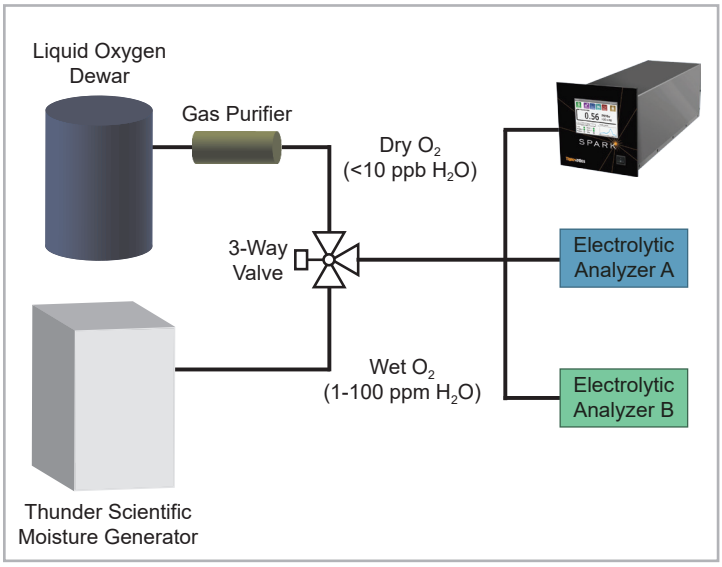

Fig. 1 Test setup for Spark H_2O validation for use with medical oxygen.

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Equipment and Sampling System

The test setup to prove equivalency for the Spark comprises the following components, as indicated in Figure 1:

1. Dry oxygen (O_2) source: boil-off gas from a liquid oxygen tank, purified to contain less than 10 ppb of H_2O
2. Wet O_2 source: variable, fixed amounts of moisture (H_2O) in O_2 are generated using a NIST-traceable Thunder Scientific Model 3900 moisture generator (5 ppm to 100 ppm H_2O)
3. Two-way switching valve to allow rapid cycling between the dry and wet O_2 sources
4. High-integrity sample lines made of electro-polished stainless steel
5. Tiger Optics Spark analyzer
6. Electrolytic Analyzer A
7. Electrolytic Analyzer B

Accuracy and Linearity Results

Using the setup illustrated in Figure 1, the Spark's accuracy and linearity over the measurement range from 5 ppm to 100 ppm were validated by performing intrusions at several different concentration levels for H_2O in O_2. Table 2 summarizes the results, indicating that the accuracy of the Spark reading, relative to the NIST-traceable Thunder Scientific moisture source, is 3.4% or better. The results in Table 2 also demonstrate excellent linearity over the measurement range. Figure 2 shows a linear fit to the Spark data, indicating almost perfect linearity with an R^2 of 0.99997.

While the laser-based Spark boasts absolute accuracy, there are some important considerations regarding the accuracy of the electrolytic-based sensors for service in O_2. Recombination takes place in the electrolytic cell when O_2 molecules from the sample gas interact with the hydrogen from electrolyzed water molecules to form additional H_2O molecules, causing a biased reading. Compensation for the recombination effect requires careful factory calibration to ensure accuracy.

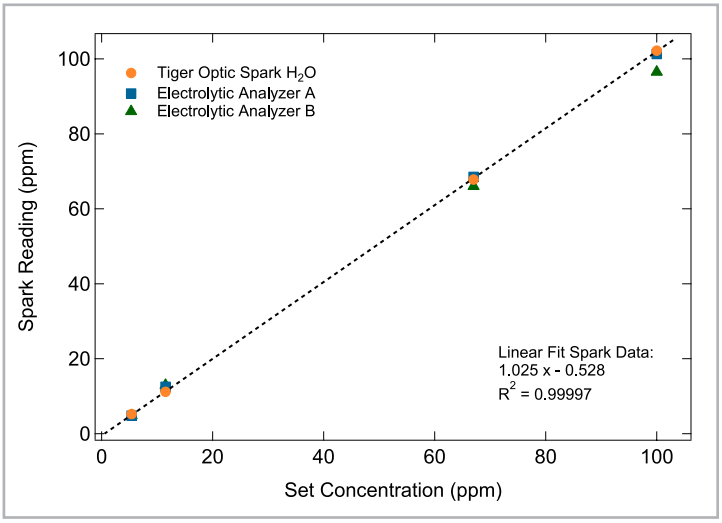

Fig. 2 Linearity comparison with vales from Table 2. The linear fit of the Spark data shows an excellent linearity factor (R^2) of 0.99997.

Table 2. Accuracy and Linearity test results summary

Set Concentration (Thunder Scientific)	Electrolytic Analyzer A Reading	Electrolytic Analyzer B Reading	Spark Reading	Spark Relative Accuracy
5.4 ppm	4.80 ppm	4.82 ppm	5.22 ppm	-3.4%
11.5 ppm	12.50 ppm	12.93 ppm	11.16 ppm	-3.0%
67.0 ppm	68.5 ppm	66.0 ppm	67.8 ppm	+1.1%
100.0 ppm	101.3 ppm	96.5 ppm	102.2 ppm	+2.2%

Speed of Response Comparison

To measure the speed of response, the three-way switching valve (see Figure 1) is used to cycle between dry gas (<10 ppb H_2O) and wet gas (67 ppm H_2O), as shown in Figure 3. Due to the very small internal volume of the sample cell and rapid measurement speed, the Spark exhibits much faster response times for both wet-up and dry-down compared to the traditional electrolytic-based analyzers. Especially on dry-down, the electrolytic analyzers lag the Spark significantly. While the Spark demonstrates a T95 response (dry-down to <3.4 ppm) of under 2 min, Electrolytic Analyzer A shows a T95 time of 11 min, while B even takes 25 min for the same initial moisture level. The inset in Figure 3 highlights this fact by showing a magnified view of the dry-down section, clearly visualizing the Spark's advantage in speed of response.

This means that, when using the Spark to qualify medical gas, the user can save up to 20 min on each measured sample. It is clear that, over time, notable savings in labor cost and improvement in production throughput are achieved when utilizing the Spark for routine product qualification.

Lowest Detection Limit (LDL) Verification

Tiger Optics defines the Lowest Detection Limit (LDL) as 3σ (three standard deviations) over 24 hours of zero gas data. To

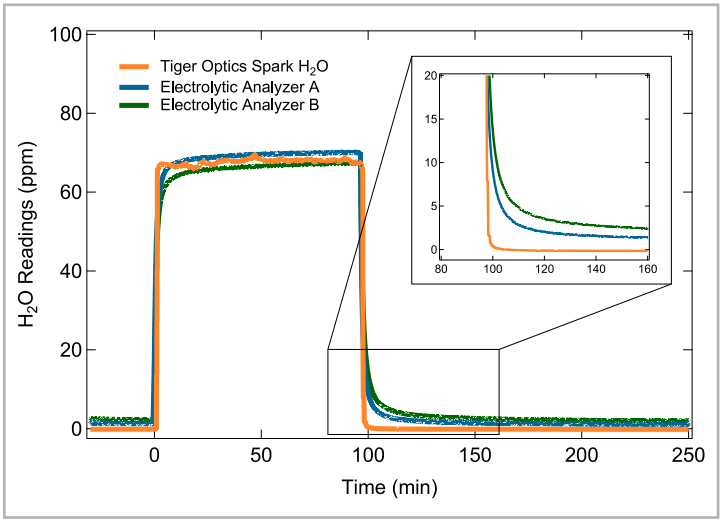

Fig. 3 Comparison of speed of response to a 67 ppm H_2O intrusion. The insert shows an expanded view of the dry-down section.

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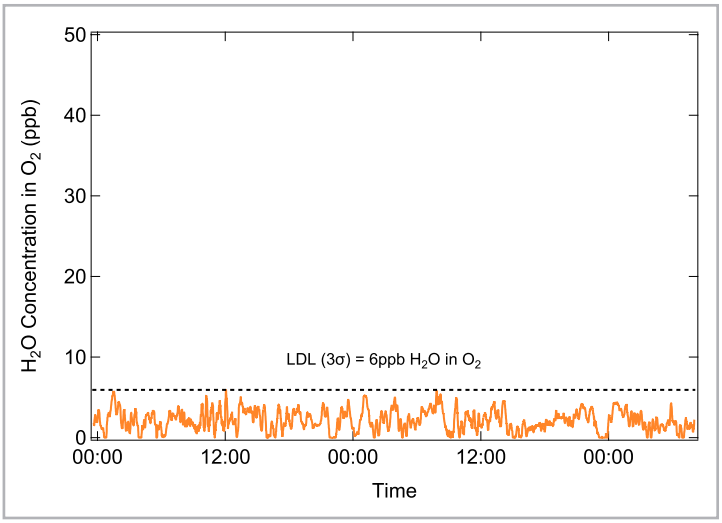

Fig. 4 Spark H_2O baseline in pure oxygen over >2 days, showing consistent readings below the specified LDL.

verify the LDL, the Spark was connected to a dry O_2 source. The baseline data is shown in Figure 4, indicating excellent agreement with the specified LDL of 6 ppb for H_2O in O_2.

As one can see in Table 1, the LDL for the typically used electrolytic analyzers is significantly higher with 1 ppm. Lower-LDL models cannot be used due to the necessity of also measuring 67 ppm for audit purposes; therefore, it is critical for these analyzers to ensure that both purge gas and sample gas are within the operating range of the analyzers, as exposure to gas drier than 1 ppm may result in cell dry-out and lead to sluggish performance. As a note, the manufacturer recommends using a Standard Moisture Addition (SMA) device or other permeation means to prevent this phenomenon. Using the Spark eliminates this problem, since it operates seamlessly at both sub-ppm moisture levels and hundreds of ppm.

Summary

The Tiger Optics Spark analyzer allows accurate measurement of moisture in oxygen to within ±4% or 6 ppb, whichever is greater, as clearly demonstrated in the present validation data. Thereby, it demonstrates equivalency with the European Pharmacopoeia standard, which mandates a relative accuracy of less than ±20%. Plus, the Spark affords a significant performance advantage over the incumbent electrolytic-based sensors, including lower detection limits, wider dynamic range, higher accuracy, and faster speed of response. This allows for better throughput and simplified product qualification, ultimately saving end-users time and money. It should be noted that the ability to conduct one-step qualification of pure oxygen for multiple applications provides significant value to users.

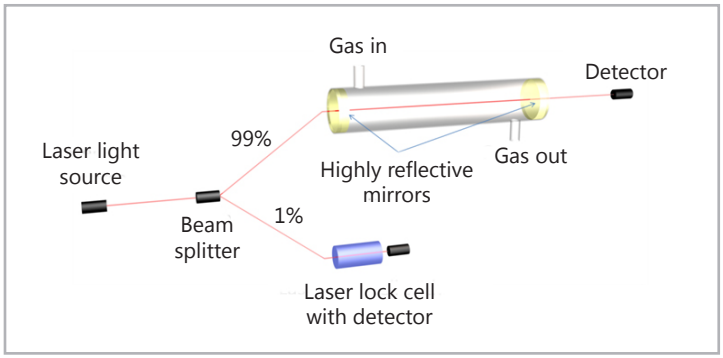

Fig. 5 Principle of Cavity Ring-Down Spectroscopy

While the results presented here focus on moisture in oxygen, the flexible Spark is also ideal for measurement in many other gases, including nitrogen, clean dry air, argon, hydrogen, and helium, without requiring a separate analyzer or different configuration.

Cavity Ring-Down Spectroscopy

All Tiger Optics instruments are based on CRDS. The key components of the CRDS system are shown in Figure 5.

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

Founded in 2001, Tiger Optics has been the preferred provider for high-performance, laser-based gas analyzers to advance industrial standards and enable cutting-edge research. By leveraging the expertise of scientists, engineers and industry specialists, we offer advanced total solutions, field support, analyzer training, and advice to help customers improve yields and reduce costs. By creating out-of-the-box solutions that deliver fast, reliable and stable measurements, Tiger Optics supports continuous innovation for gas & chemical production, semiconductor fabrication, and many other markets.

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.

References

[1]European Pharmacopeia (Ph. Eur.), 10th Edition, www.edqm.eu/en/european_pharmacopoeia_10th_edition.

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