Application Note #25

Parts-Per-Trillion Moisture Detection in Electronic-Grade Bulk Gases

HALO KA Max H_2O · HALO KA H_2O

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

This application note discusses how Tiger Optics' ultra-trace moisture analyzers help semiconductor manufacturers ensure purity of their bulk gases. We take a look at the drivers for the semiconductor industry pushing analyzer performance to new heights, and we show how Tiger's latest H_2O analyzer, the HALO KA Max H_2O, meets this challenge by reaching unprecedented levels of detection sensitivity and speed of response.

Modern Applications Propelling Semiconductor Manufacturing

The semiconductor market is seeing a new era of innovation, with new applications fueling demand for advanced semiconductor devices that put more stringent requirements on reliability, power handling capability and power consumption, while packing more functionality into a smaller package and decreasing technology nodes. Among these demanding applications are increasingly more powerful smartphones and tablets that aim—at the same time—to improve battery life. More recently, automotive sensor systems, the Internet of Things (IoT), the next generation of wireless communication (5G), and smart power grids have emerged as applications with enormous expansion potential over the coming years and decades. Future self-driving vehicles (as illustrated in Figure 1), for instance, require massive amounts of computing power to process the input from cameras and sensors in real-time; and the necessary high-performance processors must be both reliable and power-efficient.

Fig. 1 Artistic illustration of a future self-driving vehicle.

The Demand for Higher-Quality Gases and Better Analytics

To meet the challenges of these new applications, the semiconductor industry's International Roadmap for Devices and Systems (IRDS)[1] outlines manufacturing quality as one key aspect; therefore, semiconductor device manufacturers are implementing more stringent control into all aspects of the manufacturing process, from the cleanroom environment and the wafer processing tools to the raw materials used for production, many of which are gases. Consequently, improved gas quality control is one of the most important measures that are employed by semiconductor fabs to increase yields and reduce failure rates.

With the need to monitor and ensure stricter and more consistent gas quality comes a demand for more sensitive and accurate analytical technologies. At the same time, speed of response has become more important as well, as fab operators rely heavily on real-time process control to increase throughput.

Tiger Optics has been serving the semiconductor industry since its inception in 2001 with its array of laser-based moisture analyzers. In many state-of-the-art semi fabs, Tiger's Cavity Ring-Down Spectroscopy (CRDS) analyzers are the gold standard for ensuring quality of the major bulk gases that are used in the manufacturing process, which are typically N_2, CDA, O_2, H_2, Ar, and He. With H_2O as one of the most critical gas impurities, Tiger's analyzers serve a crucial role in the fab due to their ultimate sensitivity, fast speed of response, and excellent reliability.

A Sticky Situation—The Difficulty of Trace H_2O Detection

H_2O is a high priority for gas quality control in a fab, as it is very destructive to processes and one of the most difficult contaminants to avoid due to its prevalence. From an analytical point of view, moisture detection in gases tends to be a difficult task owing to the molecule's tendency to stick to surfaces. For analytical techniques that rely on physical interaction with the H_2O molecule, it is therefore very hard to achieve fast speed of response. Furthermore, detection limit requirements are stringent for semiconductor device manufacturing, ranging from low-part-per-billion (ppb) levels down to well into the

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parts-per-trillion (ppt), which is beyond the sensitivity limit of many trace detection technologies.

The most critical aspects of a successful moisture measurement for this application are:

1. Sensitivity & Accuracy of Zero Baseline
2. Baseline Stability
3. Speed of Response

Accuracy of Zero Baseline

Having a true zero is essential to ensure the analyzer's baseline accuracy. Most traditional analyzers rely on a zero gas to calibrate the instrument's baseline. But at ppt-levels of H_2O, is there really such a thing as a "zero gas"? While modern purifiers are quite effective in removing moisture, such low H_2O levels are typically below most purifiers' manufacturer specifications. Consequently, measurement techniques that rely on calibration using a zero gas can struggle to obtain an accurate zero due to the difficulty of removing H_2O completely from the "zero gas". In contrast, Tiger's CRDS systems do not rely on a zero gas measurement to obtain the instrument's zero calibration. Tiger's analyzers use a "spectroscopic zero", a wavelength where H_2O does not absorb, to determine the true measurement baseline, independent of the presence of H_2O in the gas. During measurement operation, the analyzer continuously references the concentration readings against this true zero to maintain absolute accuracy.

Baseline Stability

Another critical aspect of trace moisture measurement is the long-term stability of an instrument's zero. Most traditional analyzers experience drift in their calibration, which requires periodic "re-zeroing" of the analyzer. So what happens if the zero gas is slowly increasing its moisture content? Such a "moisture creep" can easily be caused by a failing purifier; and a slow rise in H_2O may be indistinguishable from instrument drift. In this case, the periodic zeroing of the analyzer leads to a moving offset that can mask a slow but real increase in moisture levels in your sample gas. Thus, a potential threat to your process can become undetectable.

Tab. 1 HALO KA Max H_2O and HALO KA H_2O detection specifications

	HALO KA Max H_2O		HALO KA H_2O	
Gas Matrix	Detection Limit (LDL)*	Precision (1σ) @ zero	Detection Limit (LDL)*	Precision (1σ) @ zero
In N_2	100 ppt	40 ppt	300 ppt	100 ppt
In He		10 ppt	100 ppt	20 ppt
In Ar		20 ppt	130 ppt	45 ppt
In H_2		30 ppt	200 ppt	70 ppt
In O_2		20 ppt	150 ppt	50 ppt
In CDA		30 ppt	300 ppt	100 ppt

*defined as the larger of 3σ over 24 hours or the dry-down limit

Fig. 2 Tiger Optics' state-of-the-art HALO KA H_2O trace moisture analyzer (left) and the next-generation HALO KA Max H_2O (right).

This is not the case with Tiger's systems. A direct result of the time-based and absolute nature of CRDS, any significant sources of drift, such as environmental factors or laser drift, cancel out during the measurement, making the analyzer baseline drift-free within the analyzer's LDL specification. Consequently, all real residual H_2O in the ultra-high-purity gas will still show up in the analyzer as a true reading rather than being "calibrated out".

Speed of Response

Moisture's stickiness also causes most H_2O analyzers to exhibit slow speed of response. Modern fabs, however, prefer to monitor and control their processes in real-time, which makes fast detection of impurity upsets or other issues essential. CRDS has been proven in the field to be superior in this respect because it is a purely optical, non-contact detection method that does not rely on a physical interaction with the H_2O molecule (unlike electrolytic or crystal-based moisture analyzers, for instance). Therefore, there is no interaction time that may limit the analyzer's response. Furthermore, Tiger Optics has worked consistently to improve response time by limiting internal volume and surface area, applying advanced surface treatment, and optimizing gas flow, thus offering speed of response of only a few minutes rather than hours.

These profound advantages are the main reasons that make Tiger Optics' moisture analyzers the most accurate and reliable systems to monitor the incoming bulk gases in a semiconductor fab. Consequently, the SEMI organization outlines CRDS as the standard technique for qualifying piping and measuring trace H_2O in bulk gases in SEMI F112.[2]

HALO KA and HALO KA Max—The Premium Choices for Semi Bulk Gas Monitoring

With modern semiconductor fabs seeking the highest levels of performance and reliability, Tiger Optics' premium moisture analyzers are excellent choices to consistently achieve maximum yield and throughput. Table 1 shows a summary of the detection limit and zero precision specifications for the HALO KA H_2O and the HALO KA Max H_2O.

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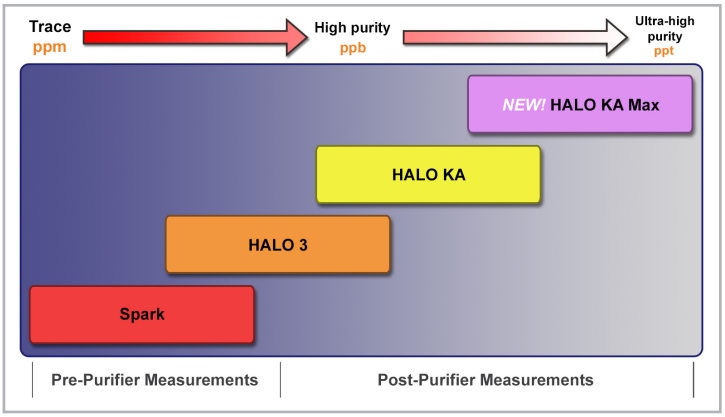

Fig. 3 Tiger Optics' lineup of trace moisture analyzers

HALO KA H_2O—The State-of-the-Art Moisture Analyzer for Modern Semi Fabs. Most advanced semiconductor fabs employ Tiger Optics' CRDS trace H_2O analyzers as the preferred technology to comply with SEMI F112 standard. The analyzer of choice for many fabs is the HALO KA H_2O (Figure 2, left). With a detection limit of 300 ppt in N_2 (specified as 3σ over a 24-hour period) and even lower in other bulk gases, the KA fits the needs of most advanced semiconductor fabs. In addition, the HALO KA saves significant space in the analyzer rack compared to the competition, since two analyzers fit within one 19" slot. In addition to high performance and reliability, the HALO KA also delivers fabs considerable savings in operating cost, with no requirements for maintenance, field calibration or spare parts.

HALO KA Max H_2O—Tiger's New Flagship H_2O Analyzer. Tiger Optics' new HALO KA Max (Figure 2, right) is an ideal choice for electronic gas makers and semiconductor manufacturers requiring detection limits for H_2O below 100 ppt. This next-generation analyzer touts both lower detection limits and faster speed of response. It is Tiger Optics' first moisture analyzer that offers a detection limit of 100 ppt (3σ/24h or dry-down limit) in all of the major semi bulk gases. This new performance benchmark makes the HALO KA Max the new flagship among Tiger's line of trace moisture analyzers (shown in Figure 3).

To illustrate the HALO KA Max's excellent baseline performance, Figure 4 shows a reading in dry N_2 over an 18-hour timeframe (black trace). The measurement demonstrates the HALO KA Max's capability to measure comfortably below 100 ppt, which actually challenges the ability of most sampling systems to achieve such a low H_2O level. Even more impressive is the analyzer's precision at near-zero levels (measured as 1σ), which is only 12 ppt in this example. The HALO KA Max's guaranteed specifications call out a precision at zero of 40 ppt in N_2 and as low as 10 ppt in He. For comparison, the red trace shows the baseline performance of the HALO KA, which also demonstrates excellent performance well below 1 ppb, exhibiting a measurement precision (1σ) of 74 ppt with a guaranteed precision specification of 100 ppt in N_2.

The key to the HALO KA Max's excellent detection limit is its entirely new electronic and optical platform, allowing the analyzer to operate with lower measurement noise. In addition, however, the new platform has more computing power, an optimized flow path, and improved wetted materials, allowing also for enhanced speed of response. This is important for preserving the analyzer's real-time detection capability when approaching low-ppt H_2O levels. Moisture dry-down time increases exponentially with lower concentrations. At some point, the analyzer's speed of response would become too slow to be useful for real-time process control. The HALO KA Max's optimized design allows it to exhibit a speed of response at ppt to ppb levels that other instruments only achieve at much higher concentrations. Figure 5 illustrates this fact impressively by simulating a process control situation with a 1 ppb control limit and occasional H_2O spikes of only a few parts per billion (denoted by the orange markers). Not only does the HALO KA Max's sensitivity pick up even the smallest spike

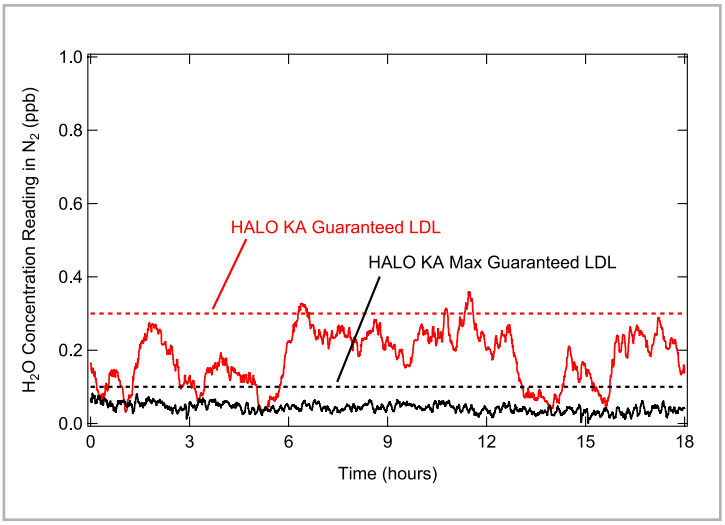

Fig. 4 HALO KA Max baseline reading in dry N_2 over 18 hours showing consistent performance below 100 ppt of moisture.

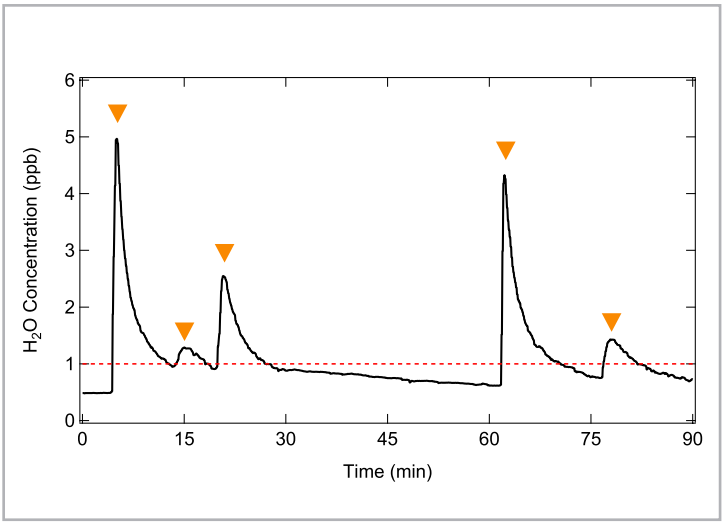

Fig. 5 Demonstrating the HALO KA Max's response to moisture spikes at low ppb levels with a simulated process control limit of 1 ppb.

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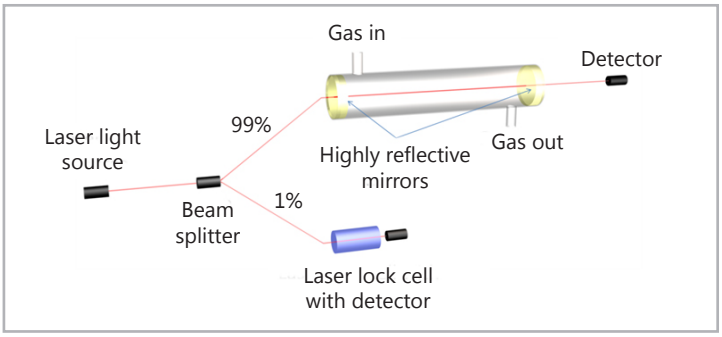

Fig. 6 Principle of CRDS

without any ambiguity, but its incredible speed of response at such low levels also instantly displays the upset. This means that your process can be protected immediately from the excess moisture. Once the H_2O source is removed, the analyzer also returns back to below the control limit within minutes, so the process can resume with minimal delay. While the HALO KA Max performs this exercise with considerable ease, it should be noted that most other analyzers would neither have the sensitivity nor the speed to respond these simulated moisture upsets with the same kind of precision.

Conclusion

In summary, Tiger's current portfolio of ultra-trace moisture analyzers allows modern semiconductor fabs to protect their processes from upsets in their gas quality more efficiently and reliably than ever before. By employing the HALO KA H_2O and the HALO KA Max H_2O, fabs are ready to meet the quality demands for present and upcoming high-performance devices.

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

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.

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References

[1]IEEE Standards Association, International Roadmap for Devices and Systems (IRDS), https://irds.ieee.org/

[2]SEMI International Standards, SEMI F112-0613 - Test Method for Determination of Moisture Dry-Down Characteristics of Surface-Mounted and Conventional Gas Delivery Systems by Cavity Ring Down Spectroscopy (CRDS), http://ams.semi.org/ebusiness/standards/SEMIStandardDetail.aspx?ProductID=211&DownloadID=3085.

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