Measuring Humidity

Breaking the Parts Per Billion Barrier

A new optical hygrometer has been developed specifically for measurement of parts-per-billion concentrations of moisture in gases. Employing a fundamental automatic chilled-mirror measurement technique, the hygrometer gives transfer-standard levels of accuracy and repeatability. Applications include metrology, calibration laboratories, and suppliers and users of ultra-high-purity gases.

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Moisture measurement continues to present designers of instrumentation with interesting challenges. For many years the moisture measurement industry has concerned itself principally with humidity ranges of –75°C dew point and upwards — in other words, moisture levels of greater than 1 ppm. The sub-ppm range was seen as extremely specialized, requiring complex and expensive analytical tools.

Increasingly, however, industry is demanding measurement capability in this sub-ppm range, and in certain areas is challenging manufacturers to go to single figure parts per billion (ppb) levels and below. This is particularly true in the semiconductor industry where the increasing complexity of products, ranging from microprocessors to memory modules and hybrid devices, demands ever greater purity of materials. Producers are simply demanding higher levels of purity in the feed gases used for processing, purging and etching the materials; and the gas suppliers have responded with calls for better and more sensitive measurement instrumentation.

To date, this has presented a dilemma for hygrometer manufacturers. Fundamental reference standards to operate below 1 ppm simply don't exist. There are, however, some reference systems using dilution, recombination and other similar techniques, but these are few and far between. As a result, there is considerable lack of understanding of exactly what is and is not possible in terms of measurement uncertainty and repeatability at ppb moisture levels. Many instrument manufacturers have simply extrapolated the operation of existing tertiary instruments such as capacitive, electrolytic and quartz-crystal hygrometers. In reality, however, instrumentation capable of measuring these moisture levels, for example the APIMS (atomic pressure ionization mass spectrometer), can cost hundreds of thousands of dollars, rendering it impractical for the majority of potential users on cost grounds alone.

OPTICAL HYGROMETERS

Clearly there is a need for a solution to this problem — an instrument with a high degree of reliability and measurement confidence, but at a reasonable price. One solution is the optical (chilled mirror) hygrometer. This measurement technique is based on the fundamental principle of obtaining, for the gas in question, the temperature at which the water vapor pressure is saturated — i.e., its condensation temperature or dew point (called dew point even if it is below 0°C). Manual hygrometers which achieve this objective have existed for more than a century. The Regnault hygrometer, dew cell, and dew cup all depend on cooling a polished surface by bubbling air through ether, adiabatic expansion of a gas, dry ice, or other means. The onset of the formation of dew or ice is detected with the human eye, and the temperature of the surface gives the dew point. The method is adequate, if labor intensive, for relatively wet gases. However, it becomes increasingly less accurate the drier the gas becomes because of the difficulty in detecting the reduced ice

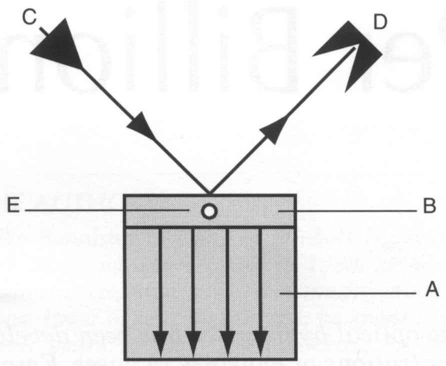

FIGURE 1. Principle of operation of an optical hygrometer with Peltier cooling.

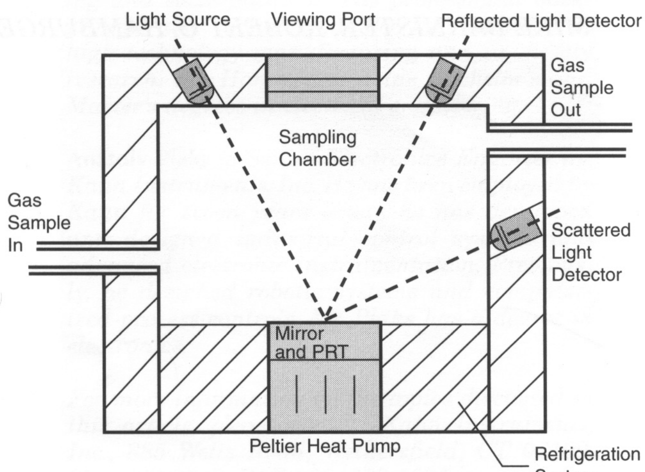

FIGURE 2. Series 4000 sensor principle.

layer with the human eye. These manual methods cannot be used below –40°C dew point without seriously overshooting the reading and, even at dew points as high as –20°C, require considerable skill and operator patience to get a reliable result.

Instruments which automate the fundamental principle have been built since the 1950s. A French system using a simple light source and photodiode, with continuous cooling by liquid nitrogen, gained some early success. The British Royal Air Force built several instruments using early thermoelectric Peltier heat pumps for wind tunnel monitoring work. The Peltier heat pump is essentially a semiconductor junction device which uses a reverse thermocouple effect — an applied current causes heat transfer across the device. This method of measurement did not gain widespread use until the 1970s with the advent of improved heat pumps and control electronics. A modern single-stage Peltier can generate a 50°C temperature differential between its top and bottom surfaces. A three-stage cascaded device can achieve a 90°C differential.

The principle of operation can be described with reference to Figure 1: (A) is the Peltier heat pump; (B) is a polished, solid copper, rhodium-plated mirror bonded to the cold side of the Peltier heat pump; (C) is an LED light-source; (D) is a photodetector; and (E) is a platinum resistance thermometer. With a dew- and frost-free mirror, virtually all the light from the LED is reflected back into the photodetector. The photodetector and Peltier device work in a closed-loop arrangement such that dew or frost on the mirror surface modulates the reflected light arriving at the detector. Suitable control electronics receive the detector output and regulate the current to the Peltier device to provide more or less cooling in the mirror. The system works in a continuous balance with a thin film of dew or frost on the mirror surface, and provides a continuous dew-point reading. The relationship between dew point and moisture content is well understood, and well documented in published literature.

The method is fundamental in nature and its accuracy depends only on the control sensitivity and the accuracy of the temperature measurement. Modern control methods allow extremely sensitive temperature control, and the use of a platinum resistance thermometer with precision measurement circuitry gives excellent temperature measurement accuracy across a wide temperature range.

Traditionally, optical hygrometers have been excellent for environmental monitoring at moisture levels in the hundreds or thousands of parts per million. However, they have been more difficult to apply to trace humidity levels due to various factors, including the need for a relatively large sample volume, low flow rate, the limitations of cooling mechanisms for the mirror, and the fact that at –100°C dew point there are approximately 12 molecules of water vapor for every 1,000,000,000 molecules of dry gas. At this level, the formation of a detectable layer of condensation can take an extremely long time with traditional optical techniques.

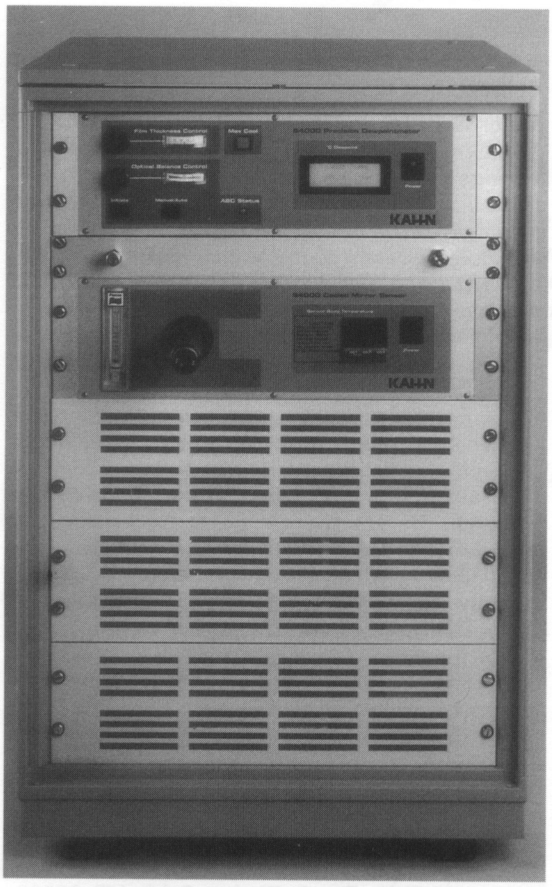

FIGURE 3. The Kahn Series 4000TRS optical hygrometer.

An effective solution is available with the development of an optical hygrometer specifically for the ppb moisture range. In addition to measuring moisture levels down to single figure parts per billion, the optical hygrometer is also practical, reliable and affordable.

OPTICAL SENSITIVITY

In essence, this new hygrometer utilizes a highly developed dual optical system which gives a significant increase in sensitivity compared with more traditional optical hygrometers. The optically tuned light source reflects from a precision coated copper mirror into a matched optical detector when no condensation exists on the mirror surface. When condensation starts to form, the intensity of the reflected light reduces marginally and some light is spectrally scattered by the water droplets or ice crystals.

A sample of this scattered light is gathered by a second detector, shown in Figure 2, and the two detected light signals are measured in a differential mode, eliminating the background level and giving a much greater signal response than in a normal single optical mode of operation.

A key requirement of successful measurement at ppb moisture levels is to minimize the sites at which water molecules can reside. The response speed of a system is often mistakenly thought to be solely a function of the sensing element, but in reality adsorption and desorption of moisture from the materials of the sampling system and sensor chamber probably have a greater overall effect. Kahn's Series 4000TRS (Figure 3) optical hygrometer is designed to minimize both the surface area and the volume of its internal parts and is manufactured from acid-etched stainless steel and PTFE. The resultant performance of the unit is ideally suited for the demanding requirements of high purity gas producers and users.

While there exists no fundamental standard below 1 ppm to which optical hygrometers, as a generic technology, can be referenced, measurement uncertainty will be extremely difficult if not impossible to validate. However, the instrument has been validated against national standards down to the low ppm levels and achieves an uncertainty of tenths of a °C dew point. Given the added complication of extending operation to low ppb levels, it would be prudent to widen this tolerance band. Nevertheless, theoretical calculation, platinum resistance thermometer calibration data and practical evaluation suggest that a measurement uncertainty of better than ±1°C is achievable at a 95% confidence level. □

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