Proxy Gas Calibration Verification Procedure for Reactive Inorganic AMC Gases:

HCl (CH_4), HF (O_2), NH_3 (CO_2)

PICARRO

Simplicity. Speed. Confidence.

Picarro cavity ring-down spectroscopy (CRDS) analyzers have a reputation for unmatched performance in drift and precision over time. This is because CRDS measures ring down time instead of light attenuation, fluctuations in the output of the laser, and the efficiency of the detector are obviated. Furthermore, the analyzers use pressure and temperature sensors in a feedback loop, eliminating environmental effects on measurement accuracy. By controlling influences such as pressure and temperature, both externally and inside the optical cavity, Picarro can control and predict the characteristics of any peak at any concentration within the measurement range, with 1% or better accuracy in the measurement results even before applying a factory calibration.

Calibrations in the Picarro factory are conforming to IEC 61207-1 and SEMI C10-1109 standards. The accuracy of this calibration is reliable for over a year and certified for each analyzer with its own Final Test document package by serial number.

Problems with Calibration Verification Using Reactive Gasses

Reactive inorganic gasses may be absorbed on the analyzer and gas delivery system surfaces during calibration and verification. Common system materials (stainless steel and Teflon) will absorb/react with these molecules to varying degrees depending on the material.

Additionally, reactive inorganic gasses will chemically react with any humidity that is introduced during the calibration process. Unless the surfaces of the delivery system components are completely passivated, these effects can cause a varying loss of molecules before they ever reach the analyzer, creating a discrepancy between the concentration of the calibration gas at the source and the concentration delivered to the analyzer.

Calibration can be Difficult in the Field

Calibration for reactive gasses such as HF, HCl, and NH_3 is much more difficult in the field because reactive gasses include high analytical uncertainties, long stabilization times (to achieve passivation), and memory effects/hysteresis due to reactive gas absorption.

Reactive and Non-Reactive Gasses are Measured During Normal Operation

During normal operation, Picarro analyzers can measure multiple gasses simultaneously within a single laser scan, which incudes common non-reactive gasses (e.g., CH_4, O_2, CO_2) found in ambient air and the target gasses (HCl, HF, and NH_3) being analyzed. The analyzers use the non-reactive gasses as a frequency reference between target gas measurements, allowing the frequency axis to be continuously corrected as measurement progresses, which eliminates a source of drift.

Figure 1 shows an absorption spectrum of typical ambient air for the HCl measurement in SI3401 and SI2018 in black, with the 5 ppb of HCl shown in red.

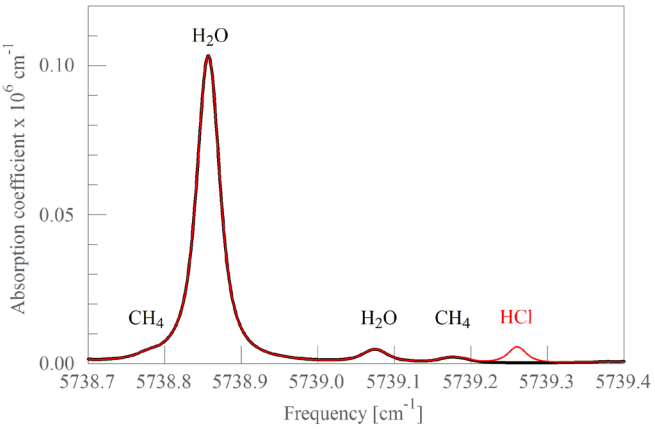

Figure 1: Ambient Air (Black) and Ambient Air with 5 ppb HCl (Red)

Advantages of Using a Proxy Gas to Verify Calibration

Sample systems and calibration setups do not adsorb non-reactive gasses such as CH_4 and CO_2. The response time of an analyzer measuring CH_4 and CO_2 (and hence the time it takes to perform a verification procedure) is only limited by the flow rate of the analyzer. Passivation and memory effects are not an issue with these gases.

Because of the issues associated with delivering reactive gases to the analyzer, Picarro does not recommend that our customers perform calibration using them in the cleanroom or at the test site. For each reactive gas, Picarro has selected a non-reactive gas that absorbs in the same spectral region and with similar intensity as the reactive gases as surrogates or "proxy" gases for the reactive gas. Picarro recommends that our customers verify calibration using these proxy gases. By using a proxy gas sample, factory calibration can be validated rapidly in the field with no need for expensive test configurations, high analytical uncertainties, long stabilization times, or memory effects due to reactive gas absorption. Measurements of proxy gases at specified concentrations allow the user to determine the validity of the calibration of reactive gas measurements by correlating equivalent losses of the two gases. *Figure 2* shows a comparison of plots for CH_4 and HCl. *Figure 2* shows a simulated comparison of 100 ppm CH_4 in Blue and 100 ppb HCl in Red. By comparing the ratio of these peak heights, it can 100 ppm of CH_4 that can be equated to about 76 ppm of HCl. This means that validating the measurement of 100 ppm CH_4 assures the user that the measurement of 76 ppb of HCl is also valid. The CRDS measurement of concentration is extremely linear and is simply a determination of absorption.

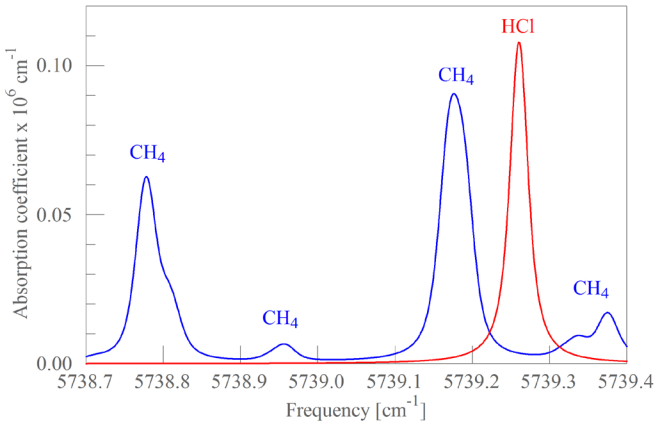

Figure 2: Comparison of 100 ppm CH_4 (Blue) and 100 ppb HCl (Red)

Because the selected proxy gases are readily available by gas suppliers and easily stored for long periods of time, this adds to their convenience. The concentration of these gases is extremely important because they are equivalent to key levels of the most important analytes. Because of this, Picarro prescribes using:

- 100 ppm CH_4 to validate HCl
- 20.95% (209500 ppm) O_2 to validate HF
- 5% (50000 ppm) CO_2 to validate NH_3

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