Application Note AN-107

May 2016

SPEC Sensor™ Characterization & Calibration Considerations

Scope

This document is provided to describe the considerations needed to characterize, calibrate, verify and validate the measurement performance of the complete system that incorporates a SPEC Sensor™ product. Sensors for Toxic gases involve measurement of a chemical property using a chemical reaction. As such the principles are guided by the kinetics and thermodynamic of reactions and sampling systems. Overall, the measurement of a gas concentration in air or any matrix gas is a “systems” property and depends on the system which includes sampling, electronics, and signal conditioning as well as the sensors themselves. SPEC Sensors offers these characterization testing services at their facilities for those that do not wish to invest in this infrastructure or have this expertise.

NOTE: *The following document provides insights into the approaches used to characterize sensor systems but is only a brief exposure to the art of metrology and gas sensors. One should consult an expert in this field in order to design systems and make measurements properly. The sufficiency of any approach or system is left to an expert. There are standards setting bodies that can define sufficiency in certain applications and the user must be familiar with the standards that apply in a given situation.*

SPEC Sensor™ Calibration

SPEC Sensors have been thoroughly characterized and are very linear over the recommended concentration range. Manufacturing is done using high volume processes and SPEC Sensors calibrates 100% of the sensors in batches unless otherwise noted in our specification. A two-point sensitivity test (0 ppm and a mid-range concentration) is performed on the sensors at the “sheet-level” under standard conditions, and a “calibration factor” (CF) is generated. The target gas concentration is typically referenced to a NIST Standard gas cylinder, when available, and the temperature and humidity measurements are done with NIST traceable instrumentation or references wherever possible. The CF is provided on the sensor label and can be used to set up the sensor but since precision and accuracy is a “systems” property, the sensor and system [electronics and sampling system and housing] should be calibrated together for best results. Especially with reactive gases, e.g. ozone, NO2, and at low levels, the calibration of the system becomes very important and applications are highly varied and often unique. Please read the label for the specific model number and product designation for this information about the sensor and its proper use.

Where the most accurate measurements are desired, SPEC Sensors recommends a calibration of the sensor in the finished device or system in which it is deployed. Electrochemical sensor responses are highly linear. This means a simple, two point calibration (zero and one concentration at the mid- or high-end of the intended measurement range) will provide accuracy across the full range of the electrochemical sensor operation. Care must be taken to calibrate sensors properly with certified gases and controlled and repeatable conditions and validate that the “system” does not degrade the sensor performance. In addition, the normal temperature dependency of the nA/ppm Sensitivity specification has been characterized for each gas sensor product. The typical effect on baseline and span are provided in each sensor product Specification Sheet. These coefficients are the average for a representative sample of sensors, however there is some variability between sensors, as indicated by the ±1 standard deviation lines on the plots on the data sheets.

NOTE: The materials and design of the rest of the detector system may affect the temperature dependence of the measurement output. ***It is recommended that the designer perform a temperature characterization test of the finished package and modify the BL and span coefficients as needed to provide an accurate compensation of device output under varying environmental conditions.***

Gas Sensor System Basics

A gas sensor system can be quite simply described as being comprised of: the SPEC Sensor™; an analog potentiostat interface circuit; and a voltage measurement device. In a typical device, one will also include an analog-to-digital convertor (ADC); microprocessor; temperature sensor; and a communications interface to the user as well as a package surrounding the sensor that forms a sampling system.

The analog potentiostat provides an interface to the SPEC Sensor™ that converts the sensor current to a voltage under controlled potential for optimum measurement sensitivity over the concentration ranges. That is, it converts the nA/ppm response into an easy to read analog voltage. *Important considerations in the potentiostat circuit design are discussed in Application Note AN-102 and reference designs are available from SPEC and a number of suppliers and users.* The ADC converts the analog potentiostat circuit output into a digital format for the microprocessor.

The microprocessor takes the digitized sensor signal and can combine this with other sensory readings if they are available and enables temperature compensation of the output, for example, for optimum accuracy over the full temperature range. This compensated signal is then communicated to the user.

NOTE: All of the components of a measurement system can have an impact on the performance of the gas sensor system's output. Thus it is important that OEMs characterize, calibrate, verify, and validate their complete gas sensor system design for an intended application.

Definitions

STANDARD CONDITIONS: SPEC Sensor calibrations are done under standard conditions of 23 ± 3 ^{o}C, 50 ± 15 %RH, and 0.9 - 1.1 atmospheres pressure. The air velocity in the calibration chamber is typically 0.01 - 0.05 m/sec. Environmental parameters such as temperature may affect the sensitivity of the sensor, but can be easily compensated as discussed in Application Notes **AN-104 Environmental Effects**

CHARACTERIZATION: Recording and analyzing the gas system's measurement behavior across its full range of operating conditions – usually performed as a subset of "all possible conditions" chosen with a target application in mind.

CALIBRATION: Adjusting a systems behavior to meet calibration requirements (often as defined by industry standards or regulations for a given application or market)

VERIFICATION: Confirmation, through the provision of objective evidence, that specified requirements have been fulfilled. E.g. ISO 9000:2000. "proves that it does what you designed it to do"

VALIDATION: Confirmation, through the provision of objective evidence, that the requirement for a specific intended use or application have been fulfilled. E.g. ISO 9000:2000. “data proves that it does what you need it to do to meet the specification or regulation or internal requirement.”

Characterization Objective

Because any of the components of the gas sensor system could impact measurement performance, a gas sensor system (configured: on lab bench, in proto-type, during design, or in production) should be characterized to determine measurement performance and any potential impacts or improvements that are needed. Some Potential Parameters used to characterize gas sensors include:

- Linearity of the measurement by some objective measure.
- Sensitivity defined as the slope of the signal vs concentration plot.
- Noise-floor of measurement (will define the sensitivity and minimum detection limit).
- Power-On Stabilization of Baseline/Zero.
- Measurement response time.
- Cross-Sensitivities [matrix and application specific].
- Repeatability of the measurement (precision defines limit for accuracy specification).
- Temperature dependence of measurement sensitivity.
- Stability over time of zero and span [with expected fluctuations of the matrix].
- Any other parameter relevant to the particular application.

Characterization testing could guide sensor system development:

- In Early stages (***screening***): Measure a small population of a variety of system designs to select the optimum design(s) [design consists of materials, geometry/structure, and methodology.]
- In Later stages (***optimization***): Measure a large population of a single design to determine any performance variability or determine calibration/compensation factors for improving the measurement performance.
- For low (near zero) concentration measurements: Understanding the temperature dependency of the signal components, power-on stabilization, signal-to noise (S-N), and cross-sensitivities may be the some important parameters to characterize.
- For accurate measurements at higher concentrations: Cross-sensitivity, linearity, and temperature dependence of sensor sensitivity may be the important parameters.

Figure 1: Characterization Test System Example – Sealed box inside of environmental chamber capable of holding a large number of units under test.

Characterization Test System Design

To meet the characterization objectives above, an example of design and design parameters typically important in a Characterization System for gas sensor systems is as follows:

- Control and measure/record **ambient temperature** of gas sensor system over the entire range with Environmental chamber and NIST Traceable temperature sensor.
- Control and measure/record **ambient humidity** of gas sensor system over the entire range with Humidity Generator and NIST traceable humidity sensor.
- Control and measure/record the **target gas concentration** over the entire range with:
    - Calibration Gas Cylinders and dilution blenders.
    - Gas Generators and NIST Traceable Gas Monitors/Analyzers.
- Deliver to and exhaust gases from well-mixed test chamber having:
    - Safety hoods and exhaust systems and scrubbers for toxic gas venting.
    - Well sealed environmental & gas sensing chamber with a non-restrictive vent to an exhaust. The vent must not cause back–pressure under any flow rates to be used.
    - All materials used in test system must be inert to the target gas and the matrix gas under all conditions. Corrosion reaction of water vapor and corrosive gases are common. Stainless steel, Teflon™, polypropylene or other high density plastics are typically used.
- Any test fixture is typically designed to allow measurements in conditions typical of the planned application. The chamber size, geometry and purge rate will be based on combination of # of devices you wish to test at one time, and the desired purge rate and the capacity of the gas delivery system.
    - If you are using small volume sources of standard, you'll wish to purge chamber and reach steady-state concentration rapidly so as not to waste expensive gas standards.
    - If you wish to measure the response time of the sensor, the chamber must respond to step-change in concentration faster than the anticipated response time of sensor and must have low dead volume or very fast purge rates.
    - To increase rate of mixing and uniformity
        - Mixing fan and baffle or multi-point injection and vent manifold to provide well-mixed chamber atmosphere.
        - Inject "pulse" of higher concentration gas, calculated volume and concentration to provide "step change" in concentration.
- Record the gas sensor system output measurement during test, typically with PC-based DAC.
- Optionally control and measure/record the **ambient pressure** of the gas sensor system over the entire operating range and time.

NOTE: *The design of characterization test systems with toxic gases and/or reactive gases is an art. Each gas and mix should be researched to understand considerations for its safe use and accurate application at ppm and ppb levels. One should consult an expert in this field in order to design systems and make measurements properly. The sufficiency of any approach or system is left to an expert. There are standards setting bodies that can define sufficiency in certain applications and the user must be familiar with the standards that apply in a given situation.*

Characterization Test Procedure

For Characterization Testing in an example we present here, one might:

- Perform gas mixture exposures of the target gas and matrix under constant T and RH, in step concentrations from Zero to Full Range and back to Zero:
    - With each gas step lasting as long as required to allow concentrations and response of system to stabilize.
    - With a Zero target gas exposure for as long as it takes in between concentrations to see full recovery of signals to background levels.
- Perform testing to various ambient environments (allow system to at reach steady to ensure all components are at the steady state T or other environmental condition this could take 2 hours or longer depending on system design). A typical sequence might include:
    - Measurement at the typically expected environment for T, RH and pressure.
    - Low Temperature.
    - Ambient Temperature.
    - High Temperature.
    - Ambient Temperature.

(It is instructive to run at least zero and one concentration of the target gas at ambient as a control to verify recovery after each excursion to temperature extremes. Obtaining the gas readings at the several temperatures allows measurement of the system temperature coefficient. Excursions to extremes would typically mimic expectations in the application.)

System Compensation/Calibration

After System Characterization, you may be able to use the results to make hardware or software compensation to the results to improve performance.

System Verification

In order to verify that your software or hardware compensations are valid [i.e. were performed correctly and were effective], you need to install all corrections and then repeat an exposure and test that the reading error is absent in the corrected output by typically repeating the test sequence containing the parameter variation being compensated.

System Validation

According to the definition of validation, this will occur in the lab and/or in the field and or at the user site in the application.

Gas Sensor System Production Calibration

It is possible that after Characterization and initial Compensation/Calibration development and Verification is completed, the Production verification system can be greatly simplified. The Production Calibration system should look for the aspects that vary from unit to unit of the system design or that cannot be easily addressed with software/hardware compensation.

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SPEC Sensors Testing Services for Customer Systems

992-003 FREE Testing Assessment: Estimate of number of units under test, schedule testing, assess data logging needs – a $500 value free!

992-004 Data logging Setup of Hardware & Software: Design and purchase and setup hardware and software to organize data results for easy comparison, correlation and analysis.

992-005 Single Gas, Bump Test – Room Temperature: Useful for comparing design options or debugging by testing functionality, measurement noise (used to estimate precision, LDL, and repeatability), and showing test population variability.

992-006 Single Gas, 0 to Full Scale – 0 and 3 concentrations – Room Temperature: Provides additional data for: LDL measurement, Power-On Stabilization, Linearity, Room-temperature sensitivity.

992-007 Single Gas, Full Range Temperature Characterization – Min, Max, Typ.: Provide additional data for: Temperature dependency of Sensitivity/Gain and Baseline/Zero at Minimum, Maximum, and Typical ambient temperatures.

992-008 Single Gas, Full Range Temperature Characterization – 7 Temperature Steps: Provide additional results of: Temperature dependency of Sensitivity/Gain and Baseline/Zero at 7 temperature steps for the best temperature compensation curve characterization.

992-009 Single Gas, Respiration Temperature/Humidity Simulation: Provides results appropriate for respiration measurement applications such as: Sensitivity and Baseline Dependence on transients from ambient conditions to Hot & Humidified Sample gas. Also delivers information relevant to: material selection and sample gas reactions with humidity.

992-010 Single Gas, RH Transient & Ambient Characterization: Provides results appropriate for portable devices and ambient applications by looking at: Zero/Baseline dependencies on RH Transients and RH Ambient Changes.

992-011 Single Gas Repeatability @ Room Temperature: Provides additional results/data for repeatability results at ambient temperatures.

992-012 Battery Discharge Dependency: Characterizes the effect of Battery/Supply Voltage changes from Full Voltage to Discharge by looking at 0 to mid-scale concentration repeatability over the 10 hours.

992-013 Multi-Gas Cross Sensitivity Testing: Room temperature Multi-gas Cross Sensitivity testing

992-014 Dedicated Test System Design and Build in our facility: While the build may take up to 7 weeks to happen, it ensures dedicated equipment for your testing needs. Future testing costs would be based on labor and consumed materials.

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