

GASES FOR HCl AND HF SENSOR CALIBRATION AND BUMP TESTING

This Technical Note details how to calibrate an HCl (hydrochloric acid) or HF (hydrofluoric acid) sensor-equipped AreaRAE, AreaRAE Pro, and AreaRAE Plus with low-concentration HCl and NO₂ (Nitrogen Oxide) gases.

There are two options:

Option 1 is related to the calibration and bump procedures of HCl gas, which is the native gas for the HCl sensor and a surrogate calibration gas for the HF sensor (10 ppm HCl).

Option 2 describes procedure for calibration of the HF sensor with surrogate gas NO₂ (5ppm NO₂).

Option 1 (HF Or HCl Sensor)

1. Common Issues With HCl Gas Delivery And Effects On An AreaRAE-Family HCl Sensor

HCl is a reactive gas. It is very well known that due to its acidic and polar properties, HCl can either react with components of a gas-delivery system (tubing, valves, regulators, etc.) or be adsorbed by the system. As a result, the target gas does not reach an AreaRAE's sensor in the specified concentration during a calibration or bump test. This yields inaccurate readings due to lack of proper gas delivery.

Gas regulator delivery pressure affects AreaRAE instrument readings. Electrochemical sensors are sensitive to pressure changes. Most constant-flow gas regulators provide variable excess pressure to gas-delivery systems due to residual pressure in a cylinder with calibration gas. This leads to incorrect unit calibration. Moreover, even if gas reaches a sensor with the desired concentration of HCl (for example, 10 ppm) at that excessive pressure, further readings will be different from what a bump test indicates in laboratory conditions. Another type of gas regulator, the demand-flow regulator, also suffers from inadequate gas pressure delivery (below ambient) and high HCl adsorption in its membrane and

tubing. As a result, a sensor reaches its saturation point only after 15 to 20 minutes after gas delivery starts.

2. Gas Delivery System Design And Procedure To Achieve Repeatable Readings

AreaRAE instruments are suitable to measure HCl or HF gas in the ranges of 0 to 15 and 0 to 10 ppm, respectively, but an improper gas delivery system can affect measurements in the field. RAE Systems has developed a procedure using a Teflon T-type gas delivery tube (P/N: MO2-3008-000) that equalizes gas delivery pressure to optimize calibration in the field. RAE Systems recommends following this procedure and gas delivery system design. If the calibration or bump testing procedure is not followed, RAE Systems cannot ensure proper gas delivery and operation of the system.

Recommended Testing Components

Gas in cylinder: The recommended gas is 10 ppm HCl with N₂ (nitrogen), manufactured by PortaGas.

Gas Regulator: PortaGas models 100-12P0.3 or 100-12P0.5, with 0.3 and 0.5 L/m gas delivery, respectively.

Tubing connection: It is recommended to use only Teflon tubing, and to use the RAE Systems T-type gas delivery tube that allows equalizing pressure of supply gas to exactly correspond with atmospheric field conditions.

Test Procedure

As shown in Figure 1, if steady-state response is not reached during calibration (10ppm HCl gas is used for calibration) due to lack of accurate gas delivery, it is difficult to achieve repeatable results with AreaRAE. For example, if an HCl sensor is calibrated at 2 minutes (60% of response) before reaching steady state, the AreaRAE HCl sensor will read high (false positive) during bump (calibration check) tests. If there is insufficient gas delivered due to gas adsorption in the walls of the regulator, lower readings than 10ppm (false negative) will be observed.

Calibration must be performed in a well-ventilated area or under an exhaust hood. During calibration, it is recommended to maintain the temperature around 20° to 22° C, and ambient pressure at 1% ±10% bar.

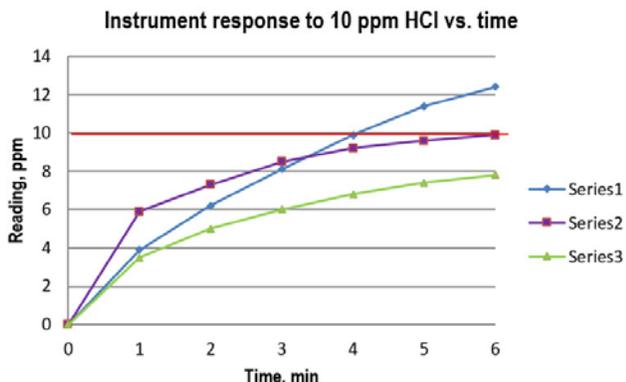


Figure 1. Results from three approaches to calibration for AreaRAE with HCl sensor.

Series 1: HCl gas delivery system was not pre-soaked for enough time before calibration but enough time before bump test. It shows an increasing reading during a bump test.

Series 2: HCl gas delivery system was pre-soaked correctly before calibration and bump test. It shows the correct reading during a bump test.

Series 3: HCl gas delivery system was pre-soaked correctly before calibration, but not before a bump test. It shows a lower reading.

3. Calibration Procedure

The calibration procedure consists of the following:

1. AreaRAE system soaking for 3 minutes (for sensor saturation). **Note:** It takes just 1 minute for the new AreaRAE Pro and Plus series.
2. Calibration time for AreaRAE is 1 minute, and for AreaRAE Pro and Plus it is 3 minutes.

Gas delivery system pre-conditioning

- Check if gas regulator is in the OFF position, and then attach it to the calibration gas cylinder.
- Remove plugs 1 and 2 on the tubing. Connect tubing to the gas regulator.
- Turn the valve on the gas regulator to the ON position for 4 min. Close plugs 1 and 2 and let the system soak for 10 minutes. The valve should be in the ON position.
- Take off plug 2 again and flush the whole gas delivery system for 4 minutes more.

- Add plug 2 and keep the gas regulator valve in the ON position.
- The gas delivery system is ready for calibration and bump (calibration check) tests.

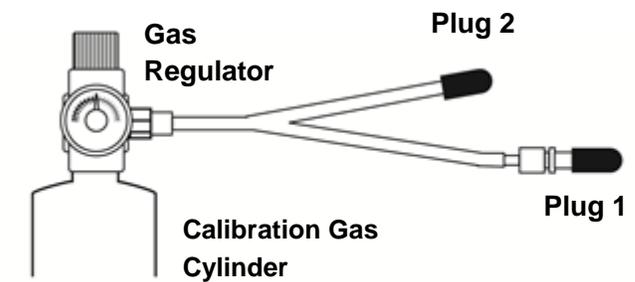


Figure 2. Fully assembled gas delivery system with cylinder, gas regulator and tubing.

Once the gas delivery system is pre-conditioned, calibration and bump testing should be started within 1 hour. If the time between gas delivery pre-conditioning and these procedures exceeds 1 hour, it may affect the proper calibration or bump test reading. In this case, a new system pre-conditioning is recommended.

AreaRAE System soaking (follows right after gas delivery system has been pre-conditioned)

- Remove plug 1 from the tubing and connect the tubing to the AreaRAE's inlet.
- Remove plug 2 (pressure equalizer) and start soaking. Let the procedure continue for 3 minutes.
- Start the calibration process (detailed in the next section).

Note: This procedure is required for the AreaRAE Pro and Plus as well, but soaking time before calibration is 1 min.

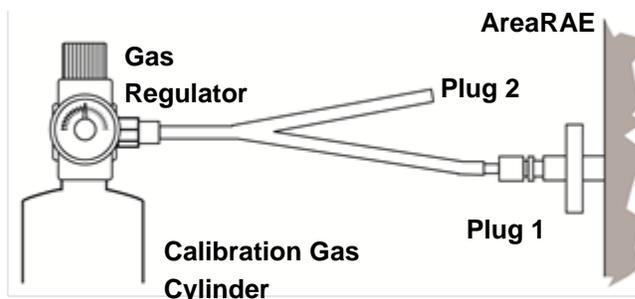


Figure 3. Attachment of gas delivery system to AreaRAE for calibration and bump testing.

Calibration procedure for AreaRAE only (follows right after 3 minutes of soaking)

- Switch AreaRAE to calibration mode (follow instructions in the User's Guide) and start calibration.
- After 1 minute, when calibration is completed, disconnect tubing from the AreaRAE and close the two hose openings with plugs 1 and 2.
- If calibration needs to be done on another AreaRAE, just repeat the **soaking** and **calibration** procedures.
- Upon finishing calibration, switch the valve on the gas regulator into the OFF position.
- Disconnect the gas regulator and tubing from the calibration cylinder and store them. There is no need to disconnect the tubing from the gas regulator.

Calibration procedure for ARAE RAE Pro and Plus

- Set the instrument to calibration mode after presoaking for 1 min as described in the section **AreaRAE system soaking** above (follow instructions in the User's Guide) and start calibration.
- Push the "Start" button to enforce calibration.
- After 3 minutes, when calibration is completed, disconnect tubing from the AreaRAE and close the two hose openings with plugs 1 and 2.
- If calibration needs to be done on another AreaRAE Pro or Plus, just repeat the **calibration** procedures.

Upon finishing calibration, switch the valve on the gas regulator to the OFF position.

- Disconnect the gas regulator and tubing from the calibration cylinder and store them. There is no need to disconnect the tubing from the gas regulator.

4. Sensor Field Verification Procedure

The sensor field verification procedure consists of the following:

1. Gas delivery system pre-conditioning.
2. Applying gas to the AreaRAE monitors and confirming sensor response.

Gas delivery system pre-conditioning

- Pre-condition the gas delivery system as described earlier.

Verification

- Remove plug 1 from the tubing and connect the tubing to the AreaRAE's inlet.
- Remove plug 2 (pressure equalizer) and apply gas. Let the procedure continue for 4 minutes, and then take a reading (all AreaRAE models).
- If the reading differs from the calibration value by more than $\pm 10\%$, perform a new calibration (follow the calibration instructions described earlier).

Note: The field verification "pass" criteria can be adjusted from 10% to a higher value, depending on the application requirements.

Option 2 (HF Sensor Only)

Through testing, it was determined that using NO₂ as the calibration gas for an HF sensor presents several benefits.

First, It does not require preliminary gas delivery system pre-soaking. Then, calibration and bump tests with it are easier, and they follow the typical procedures for calibration of EC (electrochemical) sensors. Finally, using NO₂ saves time and reduces cost of ownership: HCl gas requires 18 minutes to presoak the gas delivery system and 4 minutes to presoak the sensor and calibrate. Substituting NO₂ as the calibration gas reduces total time to 3 minutes.

The following is a suggested procedure for using NO₂ as the calibration gas for the HF sensor:

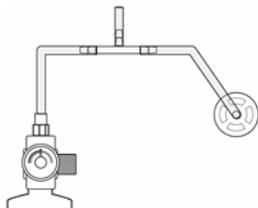
1. Span Calibration Using NO₂ For The HF Sensor

1. Press [Y/+] to select "Single Sensor Span."
2. Press [N/-] until "HF Sensor" is highlighted.
3. Press [Y/+] to select "HF Sensor."

Note: The default concentration on the instrument display is 6 ppm. Do not change it if calibration gas is 5 ppm NO₂.

4. Screw one end of a "T" using Teflon tubing to the AreaRAE Plus/Pro filter on the inlet. (You can use RAE Systems P/N: W01-3003-000).

Note: Demand Flow Regulator p/n 490-0191-000 can be used instead.



5. Attach the other end to the flow regulator of a calibration cylinder with 5 ppm NO₂ calibration gas (unless otherwise specified). The flow rate should not be less than 750cc/min ("T" type connection).

6. Start the flow of calibration gas.

7. Press [Y/+] to start calibration.

8. When calibration is complete, shut off the calibration gas flow and disconnect the calibration tubing.

9. Press [MODE] to exit to the menu. (You may perform other calibrations. Otherwise, exit.)

2. Sensor Field Verification Procedure

The sensor field verification procedure consists of applying gas to the AreaRAE monitor and confirming sensor response.

Verification

Repeat steps 4 to 6 from the previous section for verification.

Note: The field verification "pass" criteria can be adjusted from 10% to a higher value, depending on the application requirements.

Note: Freshly installed HF and HCl sensors require an equilibration time (also known as warm-up time) of at least 3 hours after installation, for the baseline to become stable enough to calibrate the sensor. Once installed, any sensor bias stays on, even when the instrument is off. Therefore, even biased sensors are ready for immediate use when the instrument is turned on again, and the equilibration time is needed only when first installed or if the battery becomes completely drained. The SensorRAE 4R+ can be used to maintain bias on sensors, so long equilibration times can be avoided when installing such sensors into a multi-gas instrument.