

Advanced Sensor Technologies, Inc.

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IMPORTANT NOTES FOR AMMONIUM ISE MEASUREMENT SYSTEM WITH 56 ANALYZER

IMPORTANT NOTE # 1:

This addendum ONLY applies to the 2nd generation Model 56 ISE Analyzers with the 2.19 software loaded. This addendum is not valid for any other transmitter models nor other 56 software versions. Note that the 2.19 software version disables all HART functionality.

IMPORTANT NOTE # 2:

This addendum ONLY covers the ISE specific aspects of the 2nd generation Model 56 ISE Analyzer with 2.19 software. For all shared functionality, refer to the main manual.

IMPORTANT NOTE # 3:

This ISE addendum assumes the basic ammonium configuration. Specifically, the pH compensation is **NOT enabled in the described use. For applications where the pH will be 8.5 or higher, please refer to the advanced configuration with pH compensation enabled.**

IMPORTANT NOTE # 4:

There MUST exist a method to perform a timely offline determine of the ammonium concentration from a grab sample near the sensor installation point. This is necessary for the critical “Standardize (grab)” calibration to synchronize the inline and offset readings.

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Confirm Correct Sensor Type & Analyzer Configuration for Planned Use

Before proceeding further, it is recommended that a review of the technical document linked below is conducted as it describes the general provisions common to all online ion selective measurements:

http://www.astisensor.com/GENERAL_GUIDE_TO_ONLINE_ISE_MEASUREMENTS.pdf

The suitable temperature range of the AB 6410 or AB 8410 ammonium ISE sensors is five to forty (5-40) degrees Celsius (41 to 104 degrees Fahrenheit). The supported pH range of the AB 6410 or AB 8410 ammonium ISE sensors is 2.5 to 11.0. A dual channel ammonium/pH configuration with the pH compensation feature enabled may be required when the pH is 8.5 or greater. If you are unsure if your application requires this dual channel mode please consult the factory for further assistance. For the characteristic pH and temperatures at most municipal water and wastewater type applications, this pH compensation described is not normally required.

At pH levels above 8.5 at the most common temperature conditions the ammonium sensor will not detect the total ammonium content, as some of the ammonium ion will be converted into the form of dissolved NH₃ gas form. Please see link below for a more detailed discussion about this topic:

http://www.astisensor.com/pH_Compensation_Total_ISE_RAI.pdf

Please contact the factory for applications requiring ammonium ion selective measurement where the pH will be above 8.5 for further assistance.

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Basic Configuration & Installation for 56 Analyzers in Ammonium Low Flow Panels

The following steps are required for an installation of a new Ammonium sensor:

- 1) Ensure that your Model 56 transmitter with 2.19 software loaded is in the proper mode and configuration. This assumes that the configuration shall be for the case where no pH compensation is required. Under the Measure Menu, the “Ammonium” choice should be selected for the channel to which the ammonium ISE sensor will be connected (see screenshot below as a guide). This screenshot below shows the sensor without an integral preamplifier, which is the most typical configuration for most inline & submersible AB 6410 sensors supplied, although this can be toggled to the preamp in sensor choice when appropriate.

The screenshot shows a 'Live display' window with a navigation bar at the top containing 'Outputs', 'Alarms', 'Measure', 'Temperature', 'pH diagnostic setup', and 'Security'. The 'Measure' menu is active, showing the following settings: 'Measurement' set to 'Ammonium', 'Show results as' set to 'Free ammonium', 'Units' set to 'ppmasNH4', 'Filter' set to '4 sec' and 'Adaptive' (with a note '* see(1)'), and 'Pre-amplifier location' set to 'Analyzer' with a blue checkmark. A 'BACK' button is located at the bottom right of the configuration area. Below the configuration area is a 'Fault/warning banner'.

- 2) Ensure that the ammonium ISE sensor is properly wired to the sensor channel that you have configured for ammonium ion measurement. Find below links to the wiring detail both for sensors with and without integral preamplifiers:

http://www.astisensor.com/Rosemount_1056_1057_56_No_Preamp_Hookup.pdf

http://www.astisensor.com/Rosemount_1056_1057_56_With_Preamp_Hookup.pdf

For convenience both of these wiring schematics linked above are included in this ISE addendum.

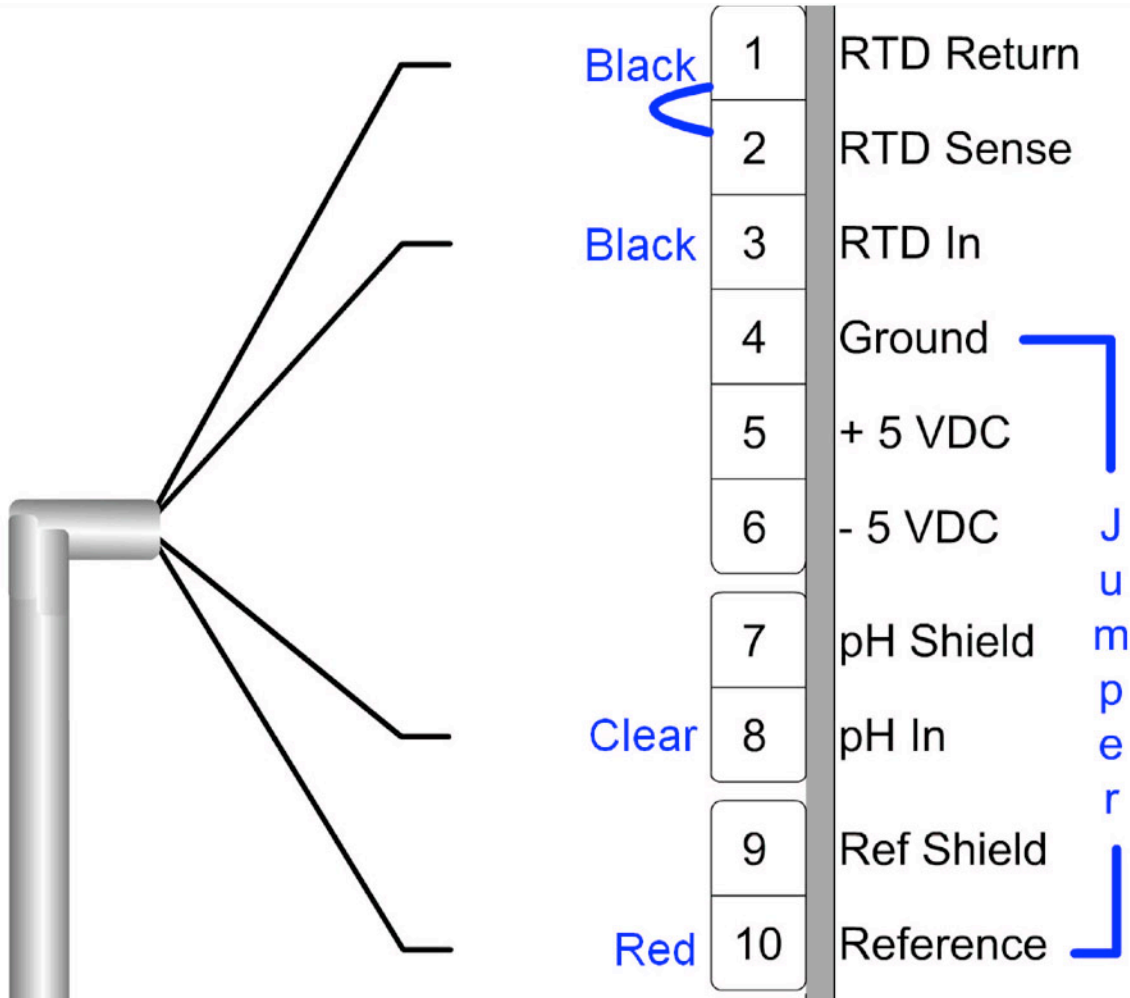
- 3) Place sensor into process and allow it to find electrochemical and thermal equilibrium. The time required for this stabilization period may vary depending upon the particular application.
- 4) To account for any differences between the presumed or used calibration standards and the actual measured solution, a grab sample should be taken and analyzed by a suitable analytical method, and the online ammonium ion selective measurement system adjusted to read the grab sample analyzed value. The sensor should be left continuously in service while this grab sample offset calibration performed. Details on the exact steps for this critical “Standardize (grab)” process offset calibration can be found later in this manual.

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Connection Diagram of Iotron™ pH / ORP / ISE Sensors **Without** Preamplifiers to Rosemount 1056/1057/56 pH/ORP/ISE Analyzers

Connection from Iotron™ Sensor to Terminal Block in Rosemount Transmitter



Note 1: The temperature compensation element is 100 or 1000 Ohm Platinum (autoswitched).

Note 2: For ORP or Ion Selective Sensors, please put the active signal (white) to terminal 8 (indicated as pH In).

Note 3: Terminals 4 & 10 and terminals 1 & 2 must be tied together to satisfy the analyzer input requirements and disable the reference diagnostic features (pH glass diagnostics should still be available).

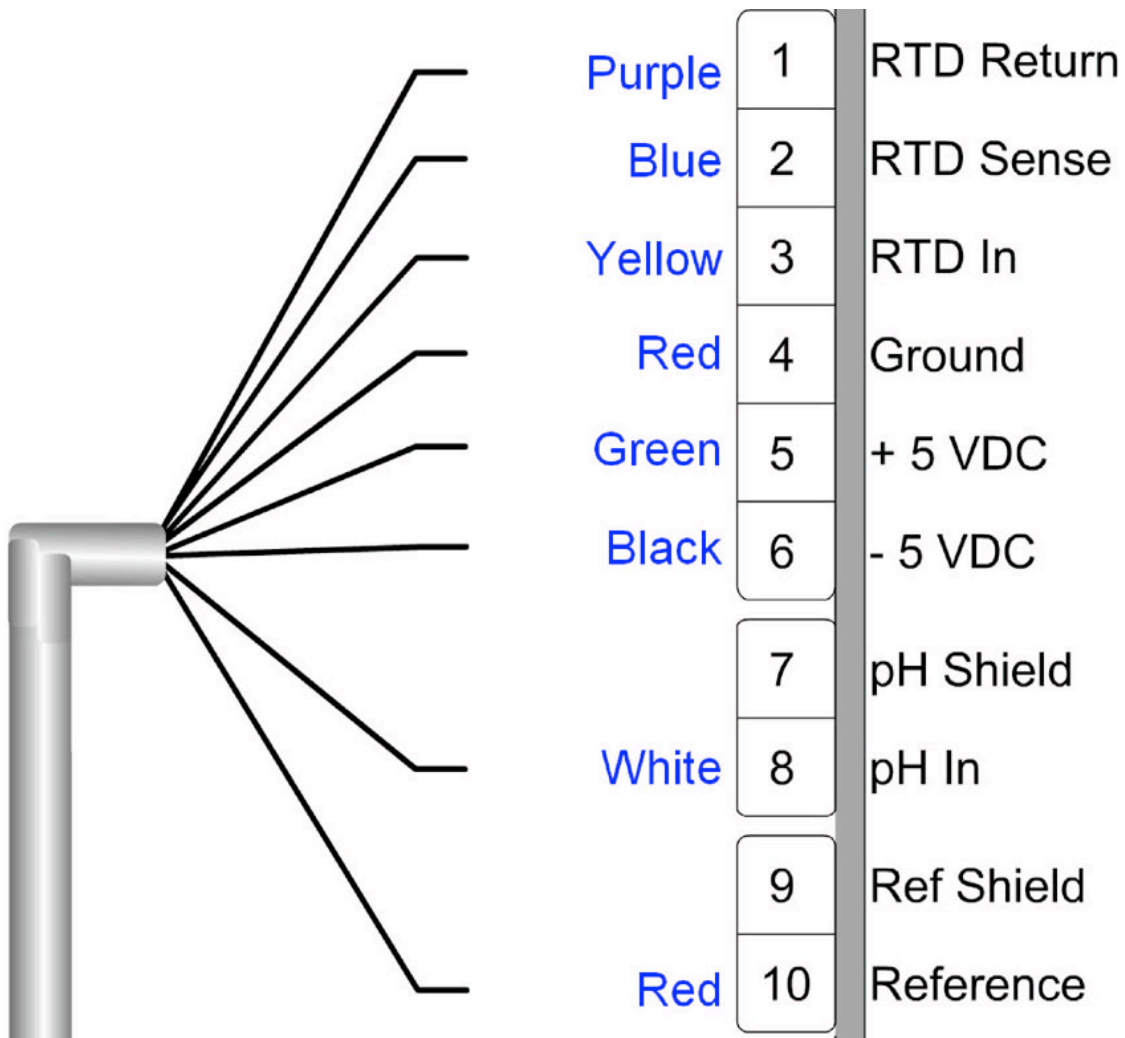
Note 4: For Dual or Triple Channel Analyzers, please ensure that the proper type of sensor is connected to the proper input board.

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Connection Diagram of Iotron™ pH / ORP / ISE Sensors **With** Preamplifiers to Rosemount 1056/1057/56 pH/ORP/ISE Analyzers

Connection from Iotron™ Sensor to Terminal Block in Rosemount Transmitter



Note 1: The temperature compensation element is 100 or 1000 Ohm Platinum (autoswitched).

Note 2: The preamplifier does not support diagnostic features (if any).

Note 3: For ORP or Ion Selective Sensors, please put the active signal (white) to terminal 8 (indicated as pH In).

Note 4: For Dual or Triple Channel Analyzers, please ensure that the proper type of sensor is connected to the proper input board.

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Grab Sample Offset Calibration (CRITICAL)

The standard (grab) calibration allows the inline ammonium sensor to be standardized in good agreement with the offline grab sampling method chosen without ever having to remove the sensor from process service. This critical grab sample offset calibration needs to be repeated from time to time as required to keep good agreement between the inline and offline readings. If the frequency with which these grab sample offset calibrations needs to be performed to keep good agreement with the offline determinations this may indicate a suboptimal installation or that the sensor is nearing its end of service and might need to be replaced.

A grab sample should be taken from the process and analyzed by a suitable method for ammonium ion concentration. There are a variety of ways to perform the grab sample analysis although the most commonly employed technology is a portable photometer when a full field lab is not available. Such portable photometers often have a full range of 0-3ppm for the lowest sensitivity model, as well as range such as 0-10ppm and 0-50ppm for measurement in higher concentrations. For measurement in solutions with higher turbidity, a one to ten volumetric dilution is recommended before performing an analysis with such a photometer. Special care should be taken if the value obtained is in the bottom 10% of the top 10% of the full scale range of any photometer since best results are typically obtained at or near 50% of the full-scale range. The grab sample determined concentration of the process sample is entered into the “Standardize (grab)” calibration as further described below. No other calibration modes should be used for entering the grab sample determined value.

VERY Important Note about “Standardize (grab)” Process Offset Calibration:

The sensor should be left in service the entire time that this calibration is performed. **The sensor should not be removed from service while performing the critical “Standardize (grab)” calibration.** Ensure that the installed sensor has been allowed sufficient time for stabilization prior to performing this calibration.

Steps for performing STANDARD (GRAB) Calibration

1. After choosing the sensor channel configured for ammonium measurement (Calibrate → S1/S2 Measurement), you will be presented with the following calibration choices (see screenshot below):

Live display

Why is calibration necessary? To find out, press INFO. Otherwise, choose the desired calibration method.

Standardize (grab) ✓

Slope/offset

Two point standard

One point standard

BACK

Fault/warning banner

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2. Once you have chosen the correct “Standardize (grab)” mode for the calibration you will be presented with the following next menu choices. Select the “Take grab sample” option:

The screenshot shows a 'Live display' window with a title bar. Below the title bar is a text area containing the instruction: 'Standardizing against a grab sample involves two steps: collecting the sample and entering the results of the analysis. You must enter test results within seven days after the sample was taken.' Below this text are three buttons: 'Take grab sample', 'Enter test results', and 'Cancel'. A 'BACK' button is located in the bottom right corner of the window. At the bottom of the window is a 'Fault/warning banner' area.

3. After entering that you have taken your grab sample, additional instructions are provided about the time correcting aspect of the Standardize (grab) calibration routine. This 2.19 software allows for a time delay of up to seven days between when the grab sample is taken and when the determined value is entered. For best results it is recommended to minimize the time delay to the minimum possible for the installation site.

The screenshot shows a 'Live display' window with a title bar. Below the title bar is a text area containing the instruction: 'Install the sensor and allow readings to stabilize. Take a sample of the process liquid and press ENTER. The analyzer captures the raw data needed for calibration and stores it for seven days pending entry of the test results. After seven days the data are erased and a new grab sample must be taken.' Below this text is a single button: 'Press ENTER to continue'. A 'BACK' button is located in the bottom right corner of the window. At the bottom of the window is a 'Fault/warning banner' area.

4. After pressing the “ENTER” key the software will store the time and date at which you took the grab sample for use with the onboard automatic correction for time induced changes to the inline readings.

The screenshot shows a 'Live display' window with a title bar. Below the title bar is a text area containing the instruction: 'Data have been taken and stored. Press EXIT to return to the main display.' Below this text is a 'BACK' button in the bottom right corner of the window. At the bottom of the window is a 'Fault/warning banner' area.

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- Analyze the grab sample taken by a suitable method in as timely a manner as possible. You will need to have this offline determined value to complete the standardize (grab) calibration process.
- Navigate back to the menu options for the “Standardize (grab)”, and choose the “Enter test results” option.

The screenshot shows a 'Live display' window with a title bar. Below the title bar is a text area containing instructions: 'Standardizing against a grab sample involves two steps: collecting the sample and entering the results of the analysis. You must enter test results within seven days after the sample was taken.' Below this text are three buttons: 'Take grab sample', 'Enter test results', and 'Cancel'. A 'BACK' button is located in the bottom right corner. At the bottom of the window is a 'Fault/warning banner'.

- A screen will appear to enter your offline determined value of the grab sample that was taken in step 3.

The screenshot shows a 'Live display' window with a title bar. Below the title bar is a text area containing instructions: 'Enter the concentration of the grab sample with the analyte expressed in exactly the same form shown in the units below. The laboratory test and the sensor may not be measuring exactly the same thing. For an explanation of how the analyzer reconciles the difference, press INFO.' Below this text is a label 'Grab sample contains' followed by a text input field containing the value '800' and the unit 'ppm as NH4'. A 'BACK' button is located in the bottom right corner. At the bottom of the window is a 'Fault/warning banner'.

- If the calibration was successful, the following screen will appear indicating the results (see screenshot)

The screenshot shows a 'Live display' window with a title bar. Below the title bar is a text area containing calibration results: 'Calibration results: Isopotential voltage = -2 mV, Change from previous = 18 mV, Calibration successful. To return to main display, press EXIT.' A 'BACK' button is located in the bottom right corner. At the bottom of the window is a 'Fault/warning banner'.

- Repeat this Standardize (grab) calibration as often as may be required to account for sensor drift over time for the given installation. The frequency with which this procedure is performed will vary from site to site depending upon a number of factors.

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Cleaning and Maintenance of

AB 6410 or AB 8410 Ammonium ISE Sensors

Cleaning is generally only recommended if the tracking of the inline ammonium ISE sensor compared to the periodic grab sample determinations diverges over the course of time. This might mean that some build-up has accumulated on the sensing tip making it less responsive to the changes in ammonium ion activity. The frequency of cleaning will depend on the nature of the process water and the extent of build up observed of on the probe tip. If the inline trending as compared to the periodic grab samples does not improve after following the cleaning procedure this may mean that the sensor is nearing the end of service life or else has been exposed to some conditions beyond its capability. Recall that the sensor DOES NOT have to be removed for the standard (grab) offset calibration.

CAUTION: Do not use any cleaning regimen that is not contained in this ISE addendum as it may damage the sensor causing shortened lifetime or even possibly render it inoperable.

CLEANING:

1. Thoroughly rinse the sensor tip with deionized (DI) water. If DI water is not available you can use distilled water instead. Gently blot the sensor tip dry with a soft tissue.
2. The ammonium sensor tip can be cleaned with isopropyl alcohol to remove any type of organic oily or waxy build-up. No other solvents or reagents should be used without contacting ASTI to ensure that it is suitable.
3. Scrape the entire reference area clean with a sharp blade. This reference is a solid-state conductive polymer and cannot be damaged with ordinary scraping of the surface with a clean sharp blade. Please take care not to gouge into the reference itself and especially **DO NOT SCRATCH THE SENSING MEMBRANE.**
4. Once the reference junction has been cleaned, rinse it thoroughly with DI water. The sensor can then be installed back into service. Sufficient time should be allowed for the sensor to equilibrate with the process water after such a cleaning regimen before performing a subsequent standard (grab) calibration.
5. Any calibration ammonium standard solution can serve as conditioning solution for extended storage. Do not allow sensor to be exposed to air for prolonged periods of time (this will cause the reference junction to become dehydrated). Always store sensor in solution when not in service in process. The cap should be filled and sealed onto sensor tip securely sealed with TEFLON tape.

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Miscellaneous Notes

- The decimal place can be moved in any screen of the analyzer by placing the cursor over the decimal place and using the up and down arrows to move the decimal point to any position.
- Do not to allow air bubbles to get trapped near the ammonium ion selective sensing membrane. The presence of even small air bubbles will cause erroneous readings and/or drift.
- The ammonium sensor is comprised of a high-impedance organic membrane system. Care should be taken not to move or touch the cable once a value is being stabilized. Touching the sensor cable can induce noise in the signal that may result in erroneous measurement values and/or calibrations.
- Please see the specification sheet and hook-up schematics that can be found in the AB 6410 or AB 8410 ammonium ISE sensor shipping box. This information is included at the end of this ISE addendum for convenience for situations whereby the original hard copy was lost or misplaced.

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2-Point Slope Calibration (OPTIONAL)

The slope of the ammonium ion selective sensor (mV per decade ion activity) is a characteristic value for the given application type. The factory-programmed slope for ammonium ISE sensors rarely needs to be adjusted for municipal water or wastewater type applications. Most municipal ammonium ion measurements can be perfectly accomplished without ever performing the 2-point slope calibration (default slope is used).

For measurement of ammonium ions applications other than municipal, better results may be obtained by performing a slope calibration with standards that mimic the expected service conditions. In most cases the background can be left as 1 Molar MgCl₂ (see page 13) and just the ppm levels are adjusted. For example, to measure samples typically from ~10 to 1,000 ppm commonly 50 & 500 ppm standards are employed to gain the suitable slope for these measurements. The aging of the sensor normally only induces only a drift in the potential of the sensor, which is corrected by the standardize (grab) calibration. Aging of the sensor does not typically induce a change in slope, even near the end of service use (the slope stays rather constant over time).

IMPORTANT CAVEATS FOR A SUCCESSFUL 2-POINT SLOPE CALIBRATION:

- No commercially available ammonium calibration standards exist that are suitable for performing a 2-point slope calibration with these ammonium ISE sensors. The procedure to make suitable ammonium ion calibration standards for municipal applications is provided on page 13.
- Rinse of sensors with DI water and blot dry before starting calibration
- Gently shake down sensor to ensure that there is not air bubble entrapped inside the sensing element
- Place the sensor at a ~45 degree angle into the standard checking that there are no air bubbles on the sensing tip. If any air bubbles are seen, gently shake the sensor to free the air bubbles from tip
- Sensor should be a thermal equilibrium before performing 2-point slope calibration
- Allow sufficient time for the reading to stabilize in the first low 2ppm standard before starting your 2-point slope calibration procedure.
- Use the low 2.0 ppm standard for the first calibration point, and then use the high 20.0 ppm standard for the second calibration point. Allow sufficient time for the reading to stabilize before proceeding to calibration to the high 20.0 ppm calibration standard.

EVEN AFTER A 2-POINT SLOPE CALIBRATION YOU MUST STILL SUBSEQUENTLY PERFORM THE CRITICAL STANDARD (GRAB) OFFSET CALIBRATION TO REFERENCE THE INLINE READING TO AN OFFLINE DETERMINED VALUE FOR THE GRAB SAMPLE FROM THE INSTALLATION LOCATION.

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After choosing the sensor channel configured for ammonium measurement (Calibrate → S1/S2 Measurement), you will be presented with the following calibration choices (see screenshot below):

Live display

Why is calibration necessary? To find out, press INFO. Otherwise, choose the desired calibration method.

Fault/warning banner

Choose the “Two Point Standard” option from this calibration menu. Follow the on-screen step-by-step instructions. A part of this procedure will involve entering the value for your first low ammonium ion calibration standard (2.0 ppm solution from page 13) followed by entering the value for your second high ammonium ion calibration standard (20.0 ppm solution from page 13). After completion of the 2-point slope calibration the slope obtained from the procedure will be reported.

The factory default value is +52.16mV per decade for the ammonium ion selective sensor slope that is characteristic for the most municipal application uses. This was obtained through extensive wet testing by mean of the much more robust and accurate standard addition technique to determine the most characteristic sensor slope. The standard addition methodology is always the most accurate method to obtain the real-world effective sensor slope for the planned field application use. If you obtain a slightly different value with your 2-point slope calibration using the ammonium ion calibration standards, this is most likely due to degradation of the standard itself, some minor suboptimal part of how the calibration procedure was performed or else just reflective of the fact that the standard addition technique for slope determination is a better method for effective sensor slope determination. The offset reported after a 2-point slope calibration is not relevant since this will change once the mandatory subsequent standard (grab) calibration is performed after the sensor has been installed into service and sufficiently equilibrated. The slope does not change when performing the standard (grab) offset calibration procedure.

REPEATED FOR EMPHASIS:

YOU MUST PERFORM A STANDARDIZE (GRAB) CALIBRATION EVEN IF YOU HAVE DONE A 2-POINT SLOPE CALIBRATION WITH AMMONIUM ION STANDARDS. PLEASE SEE THE PREVIOUS PORTION OF THIS ISE ADDENDUM FOR INSTRUCTIONS ON HOW TO PERFORM THE CRITICAL STANDARDIZE (GRAB) CALIBRATION.

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Procedures for Preparation of Ammonium Standard Solutions

Materials

Ammonium Chloride - CAS # 12125-02-9 (Analytical/Reagent Grade or better, new sealed bottle preferred)

Magnesium Chloride Hexahydrate - CAS # 7791-18-6 (ACS Reagent Grade or better)

1 Liter Volumetric Flask (one each)

2 Liter Volumetric Flask (one each)

1 mL volumetric pipette

10 mL volumetric pipette

1 liter opaque plastic bottles with air-tight sealing cap (three each)

DI Water (15 MegaOhms or higher resistivity grade)

- ENSURE THAT ALL GLASSWARE IS CLEAN AND DRY BEFORE PROCEEDING.

- THOROUGHLY CLEAN EACH VOLUMETRIC FLASK AFTER PREPARING ANY SOLUTION

- SOLUTIONS PREPARED FROM THIS PROCEDURE WILL STAY GOOD FOR 1 YEAR FROM DATE OF MANUFACTURE IF STORED IN AN SEALED, OPAQUE PLASTIC BOTTLE IN A COOL DRY LOCATION

Stock Solution Preparation Procedures:

Preparation of 1.00 Molar Magnesium Chloride stock solution (DO THIS FIRST!):

1. Measure out 406.6 grams of magnesium chloride hexahydrate.
2. Place this magnesium chloride into a 2 liter volumetric flask.
3. Dilute with DI water to 2 liter mark. Mix solution well until all magnesium chloride is dissolved.
4. Seal 2 liter volumetric flask with glass stopper.

Preparation of 1,000 ppm Ammonium stock solution:

1. Measure out 2.965 grams of ammonium chloride salt.
2. Place this ammonium chloride into 1 liter volumetric flask.
3. Dilute with DI water to the 1 liter mark. Mix solution well until it is completely homogeneous.
4. Transfer this 1,000 ppm ammonium stock solution to a 1 liter plastic bottle and label appropriately.

Ammonium Calibration Solution Preparation Procedures:

Preparation of 2.0 ppm Ammonium Standard Ion Solution

1. Draw 2.00 mL of 1,000 ppm ammonium stock solution and transfer to a 1 liter volumetric flask.
2. Dilute with 1.00 Molar magnesium chloride stock solution to the 1 liter mark. Mix solution well until it is completely homogeneous.
3. Transfer this 2.0 ppm ammonium calibration solution to a 1 liter plastic bottle and label appropriately.

Preparation of 20.0 ppm Ammonium Standard Ion Solution

4. Draw 20.0 mL of 1,000 ppm ammonium stock solution and transfer to a 1 liter volumetric flask.
5. Dilute with 1.00 Molar magnesium chloride stock solution to the 1 liter mark. Mix solution well until it is completely homogeneous.
6. Transfer this 20.0 ppm ammonium calibration solution to a 1 liter plastic bottle and label appropriately.

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