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NOTES FOR TOTAL AMMONIA MEASUREMENT SYSTEM USING 56 ANALYZER WITH pH COMPENSATION ENABLED

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 advanced ammonium configuration. Specifically, the pH compensation is enabled in the described use, which is necessary for applications where the pH will be 8.5 or higher (either in the normal usage or during excursion conditions).

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.

SUMMARY OF CONTENTS:

Last Revised July 19, 2015

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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 6410or AB 8410ammonium ISE sensors is five to forty (5-40) degrees Celsius (41 to 104 degrees Fahrenheit). The supported pH range of the AB 6410or AB 8410ammonium 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 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 NH_3 gas form. Please see link below for a more detailed discussion about this topic:

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

This ISE addendum describes the advanced ammonium configuration with the pH compensated feature enabled as required for applications where the pH is above 8.5 in the normal usage or potentially during excursion conditions. The following page shows a graph for visualization of the pH dependence for the extent of ionization of dissolved ammonia gas (NH_3) into the measured ammonium (NH_4^+) ion form. As can be noted the pH at which pH compensation should be performed is a function of the temperature of the process for this measurement. This physical chemistry phenomenon for the conversion of ammonia gas into the measured ammonium ion form is what is automatically corrected with the pH compensation feature enabled on the 56 analyzed to yield the summed "total unbound ammonia" species. Note that this pH compensation does not correct for any ammonia species that is otherwise bound (such as in monochloramine for example). As such in such nomenclature this reported sum of the dissolved ammonia gas (NH_3) form and ionized ammonium (NH_4^+) form are considered as the "total free ammonia" species meaning all species that are available for reactivity.

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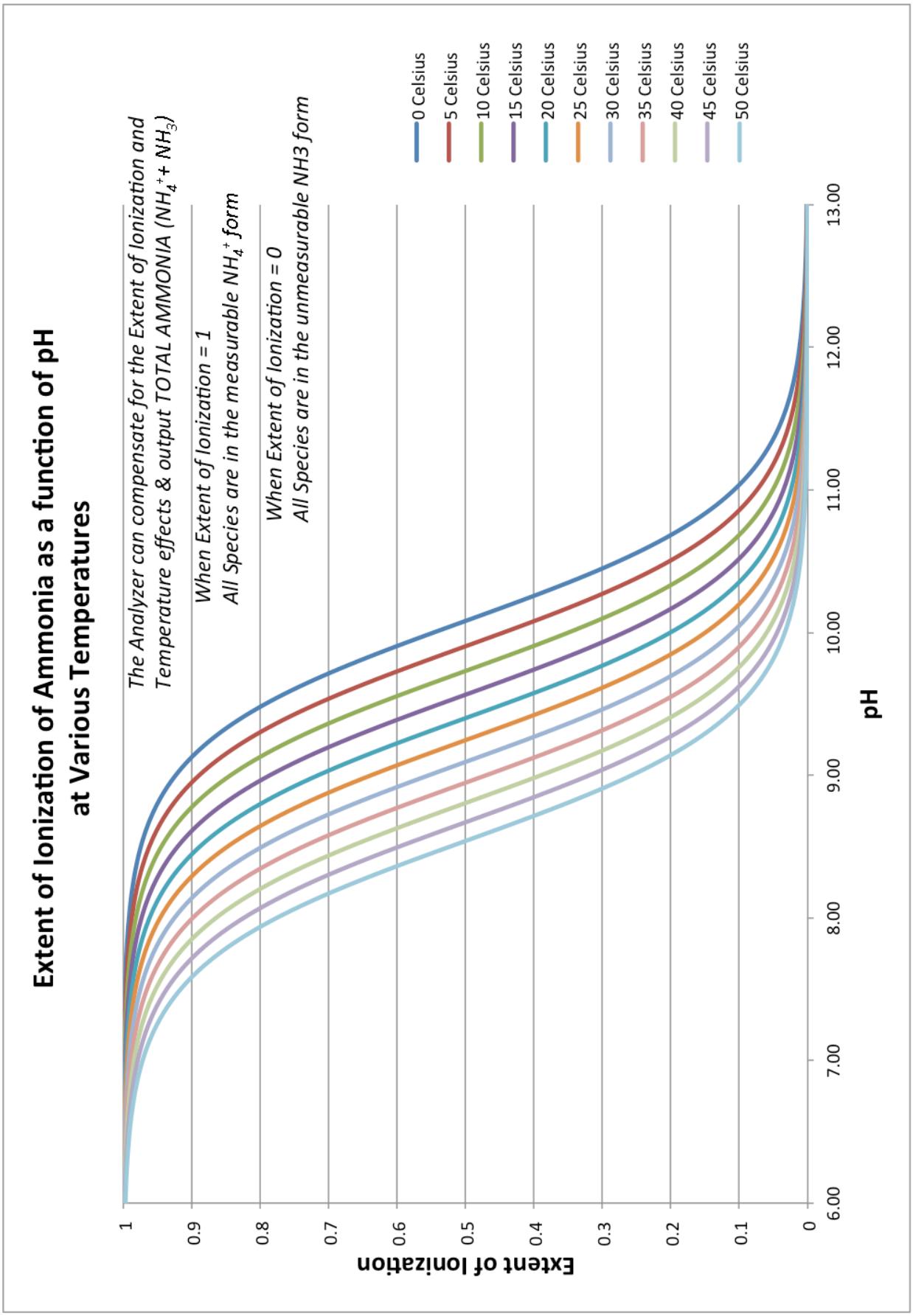

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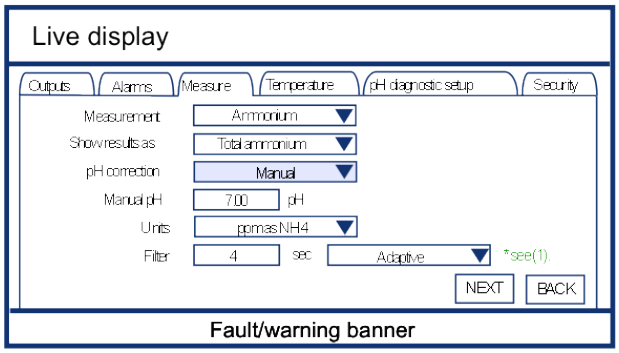
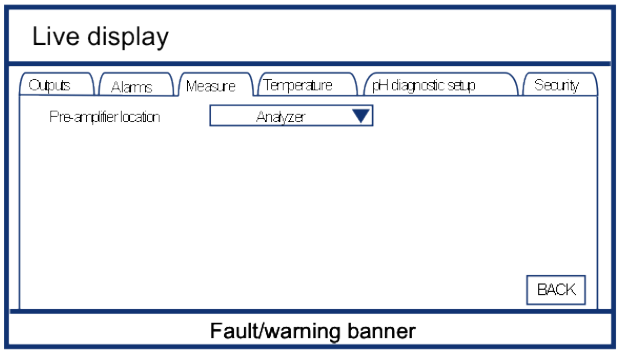
Advanced Configuration & Installation Guide for 56 Analyzers with pH Compensation Enabled for determination of "Total" unbound ammonia

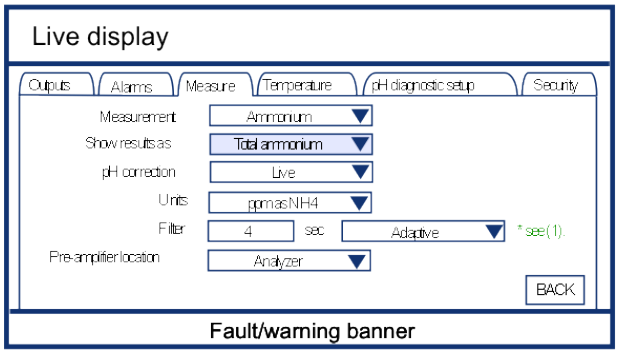
The following steps are required for an installation of a new Ammonium sensor:

1) Ensure that your Model 56 transmitter has the 2.19 software loaded before proceeding. Under the Measure Menu, select "Ammonium" for the channel 1 Measurement and "Total Ammonium" as the selection for the Show result as field. Select whether pH compensation will be perform from a manually entered value (top screenshots) or else from the live pH obtained value from pH sensor on channel 2 (lower screenshot).





Manual pH Mode (as shown in screenshots above)



Live pH compensation mode (as shown on screenshot to left)

The screenshot above shows the ammonium ISE sensor used without an integral preamplifier, which is the most typical for the AB 6410or AB 8410style sensor with no more than 20 feet of cable. This setting can be toggled to "preamp in sensor" when using a sensor that has an integral preamp to support long cable lengths, bridged lead terminations or else if an external preamplifier is employed.

2) Ensure that the ammonium ISE sensor is properly wired to the sensor channel 1 configured for ammonium ion measurement. Links are below for wiring detail for sensors with & without integral preamps:
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) Configure the second channel for pH selecting the appropriate location of the preamplifier to be used as the "analyzer" or "preamp in sensor" as is appropriate for the type of mating pH sensor to be employed. Please refer back to the main Rosemount 56 manual for this aspect of the configuration as it is entirely covered in this documentation and so there is no need to duplicate those materials here in this addendum.

4) Calibrate the pH sensor installed onto the second channel as per the documentation in the main 56 manual and as per the onscreen guide (this is normally quite sufficient even if the manual is not readily available).

5) Place both the ammonium ISE and pH sensor into process and allow it to find electrochemical and thermal equilibrium. The time required for stabilization may vary depending upon the particular application.

6) 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.

7) **Note that the 2.19 special software will automatically correctly assume that the grab sample determined value enter for the "Standardize (grab)" calibration is the total unbound ammonia species which is always characteristic for the results obtained from any offline grab sample analysis. This is a part of why it is critical that both the ammonium ion selective and pH sensors are continuously in service for the entire period of time that the "Standardize (grab)" calibration is performed.**

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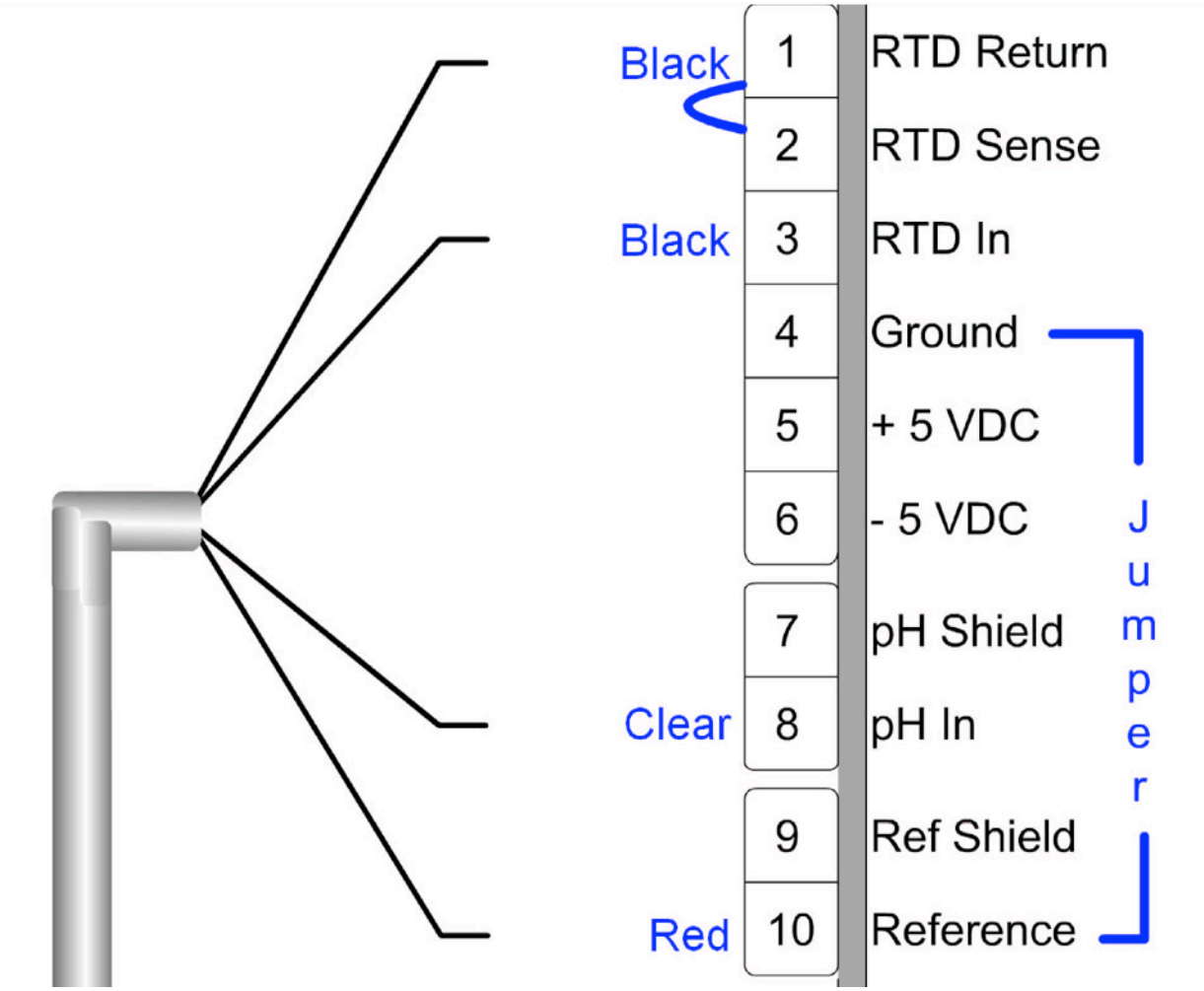
Connection Diagram of Iotron™ pH / ORP / ISE Sensors **Without** Preamplifiers to Rosemount 1056/1057/56 pH/ORP/ISE Analyzers

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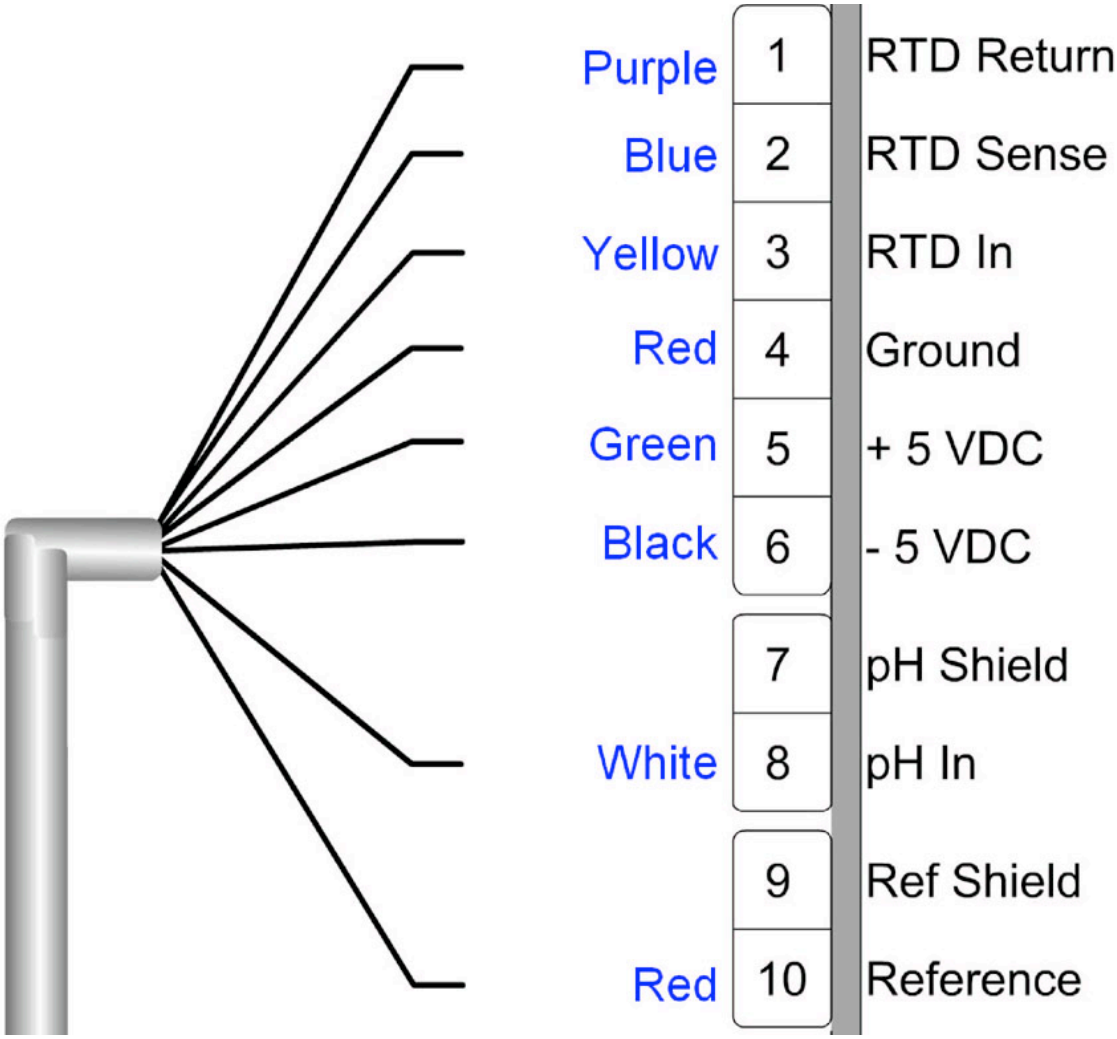
Connection Diagram of Iotron™ pH / ORP / ISE Sensors **With** Preamplifiers to Rosemount 1056/1057/56 pH/ORP/ISE Analyzers

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.

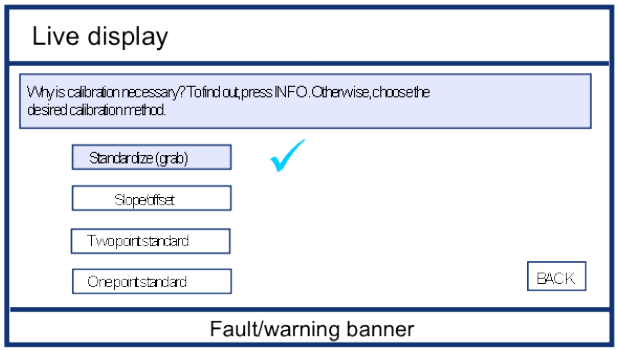
A grab sample should be taken from the process and analyzed by a suitable method for ammonium ion concentration. There are a variety of ways to perform the grab sample analysis although the most commonly employed technology is a portable photometer when a full field lab is not available. Such portable photometers often have a full range of 0-3ppm for the lowest sensitivity setting, model or mode. 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 as the uncertainty increases substantially for this situation. The best results are typically obtained at or near 50% of the full-scale range for most colorimetric type methods in cases where little or none of the potential interferences for that reagent method are present. The grab sample determined concentration of the process sample will be entered into the "Standardize (grab)" calibration as further described below. Note that no other calibration modes should be used for entering the grab sample determined value.

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

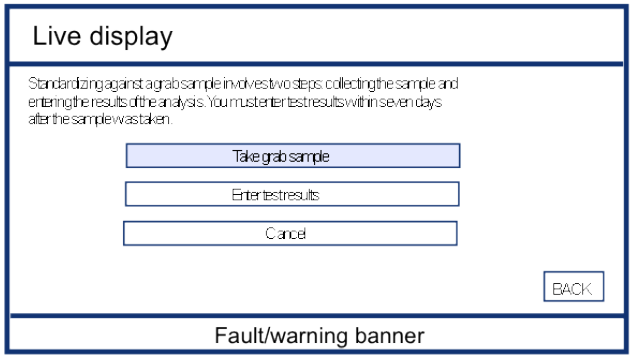
BOTH ammonium & pH sensors should be left in service the ENTIRE TIME this calibration is performed. Ensure both sensors have been allowed sufficient time for stabilization before proceeding.

Steps for performing STANDARD (GRAB) Calibration

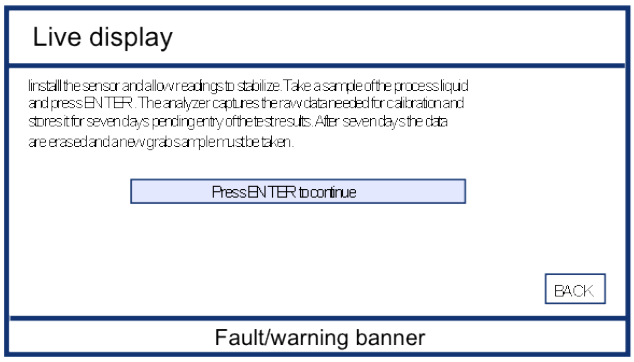
1. After choosing the sensor channel configured for ammonium measurement (Calibrate → S1 Measurement), you will be presented with the following calibration choices (see screenshot below):



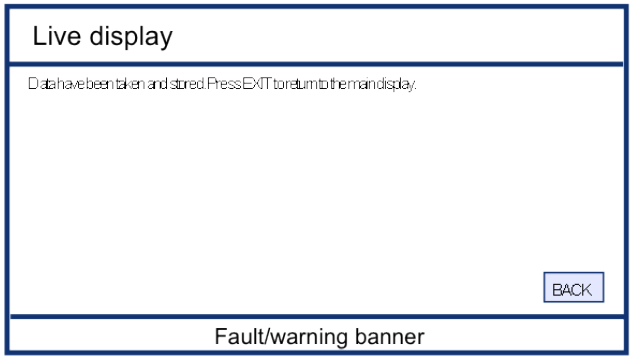
2. Once you have chosen the correct "Standardize (grab)" mode for the calibration you will be presented with the following next menu choices. Select the "Take grab sample" option:



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.

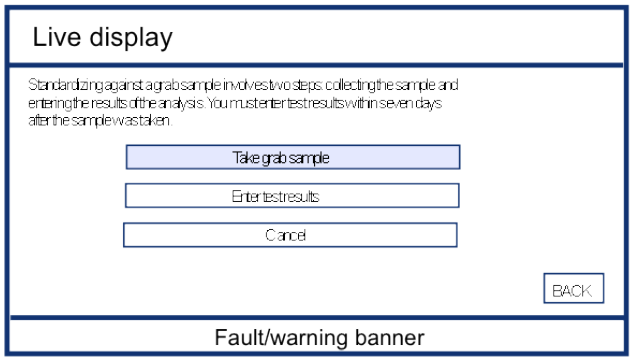


4. After pressing the "ENTER" key the software will store the time and date at which you took the grab sample for use with the onboard automatic correction for time induced changes to the inline readings.

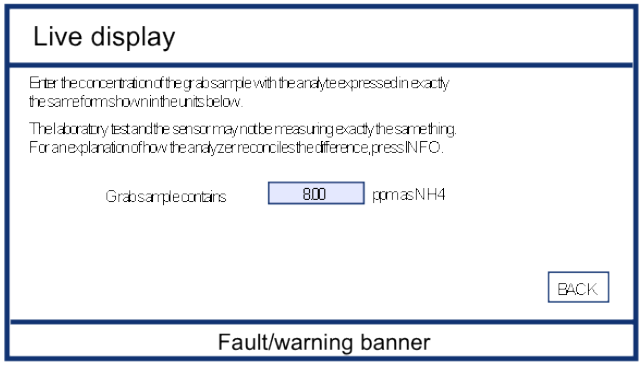


5. Analyze the grab sample taken by a suitable method in as timely a manner as possible. You will need to have this offline determined value to complete the standardize (grab) calibration process.

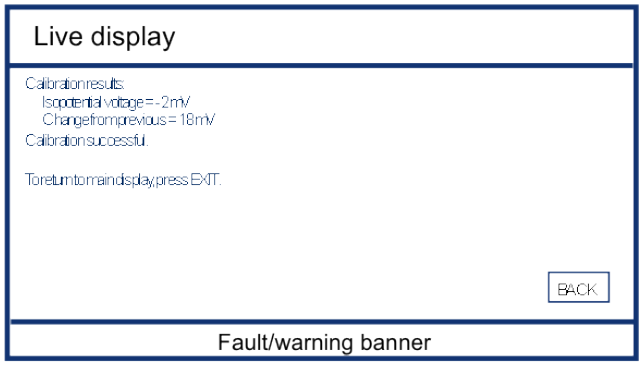
6. Navigate back to the menu options for the "Standardize (grab)", and choose the "Enter test results" option.



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



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



9. 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 6410or AB 8410Ammonium 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. It DI water is not available you can use distilled water installed 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 6410or ISE-NH4-TL-U5001ammonium 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)

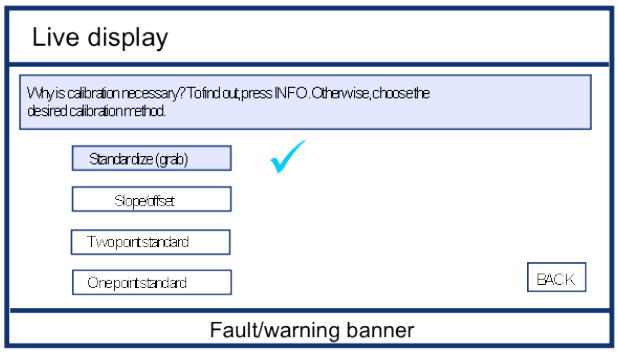
The slope response of the ammonium ion selective sensor (mV change per decade ion activity change) is a rather characteristic value. The factory-programmed slope for ammonium ISE sensors rarely needs to be adjusted. The vast majority of inline ammonium ion measurement applications can be perfectly accomplished without ever performing the 2-point slope calibration at all (factory default slope is left alone). Only the critical standard (grab) process offset calibration needs to be performed periodically as may be required and the sensor cleaned (if necessary). The aging of the sensor normally only induces only a drift in the absolute 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 very 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 the purpose of performing a 2-point slope calibration with these ammonium ion selective sensors. The ASTI factory recommended fabrication procedure is provided on page 15 of this ISE addendum. This contains the exact bill of materials and procedure to prepare ammonium ion calibration standards that are appropriate for performing 2-point slope calibration with these ammonium ISE sensors.
- 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 SUBSEQUNETLY 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.

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



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 15) followed by entering the value for your second high ammonium ion calibration standard (20.0 ppm solution from page 15). 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 most characteristic for this application use. 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 pippete
10 mL volumetric pippete
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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