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NOTES FOR FLUORIDE (TOTAL HF) MEASUREMENT SYSTEM USING 56 ANALYZERS WITH pH COMPENSATION ENABLED

IMPORTANT NOTE # 1:

Addendum ONLY applies to Model 56 ISE Analyzers with 2.19 or 2.30 software loaded. This addendum is not valid for any other transmitter models nor other 56 software versions. Note that the 2.19 or 2.30 software version disables all HART functionality.

IMPORTANT NOTE # 2:

Addendum ONLY covers the ISE specific aspects of the Model 56 ISE Analyzer with 2.19 or 2.30 software. For all shared functionality, refer to the main manual.

IMPORTANT NOTE # 3:

This ISE addendum assumes the advanced fluoride configuration. Specifically, the pH compensation is enabled in the described use, which is necessary for applications where the pH will be 5.5 or lower (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 fluoride 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 September 24, 2019

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Web Site: http://www.astisensor.com Technical Support: http://www.astisensor.com/cgi-bin/ttx.cgi

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Confirm Correct Sensor Type 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 ISE addendum entirely assumes that the fluoride ion selective sensor employed will be the special model AB 6100 type and that the pH sensor employed will be the high HF resistant PNHF 6431 model type. The suitable temperature range of the AB 6100 fluoride ISE sensors is five to seventy (5-70) degrees Celsius (41 to 158 degrees Fahrenheit). The supported pH range of the AB 6100 fluoride ISE sensors is 0 to 9.5. The typical pH usage range of 0 to 6 pH is shown on the AB 6100 specification sheet but it can be used with a pH as high as 9.5 continuously. A pH greater 9.5 will cause some premature aging on the measuring crystal itself although it can be used successfully with a pH of up to 11.0 (intermittently) if required. The AB 6100 can be used anywhere within this range of pH with equally good results. The AB 6100 type fluoride ISE sensor is the ONLY model that can be acid cleaned. The use of acid cleaning can be quite critical to soluble various fouling and build-up on the sensor that cannot be removed with this acid cleaning regimen. With this in mind it may be quite desirable to use this type of AB 6100 sensor even if the pH will rarely if ever fall below the 5.5 level during the field process conditions. If the pH does goes below 5.5 for the online use the advanced configuration with the pH compensated feature enabled is critical to ensure proper real-time determination for all unbound fluoride species occurs for proper calibration and field results.

The AB 6100 type sensors should ONLY be used for immersion/submersion installation schemes. More specifically the AB 6100 type sensor should NOT be used for any inline installation scheme. At pH levels below 5.5, the fluoride sensor will not detect the total fluoride content, as some of the fluoride ion will be converted into the form of dissolved HF gas form. The cases when the pH shall be below 5.5 will require the special AB 6100 type fluoride ISE sensors and a model PNHF 6431 HF resistant pH sensor as well as a special advanced configuration of the dual channel 56 analyzer such that calibration, display and output will be in the pH compensated "Total" unbound fluoride units (see link below for a more detailed discussion):

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

This ISE addendum describes the advanced fluoride configuration with the pH compensated feature enabled as required for applications where the pH is below 5.5 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 HF acid into the fluoride ion form. This physical chemistry phenomenon is what is automatically corrected with the pH compensation feature enabled on the 56 analyzed to yield the "total unbound fluoride".

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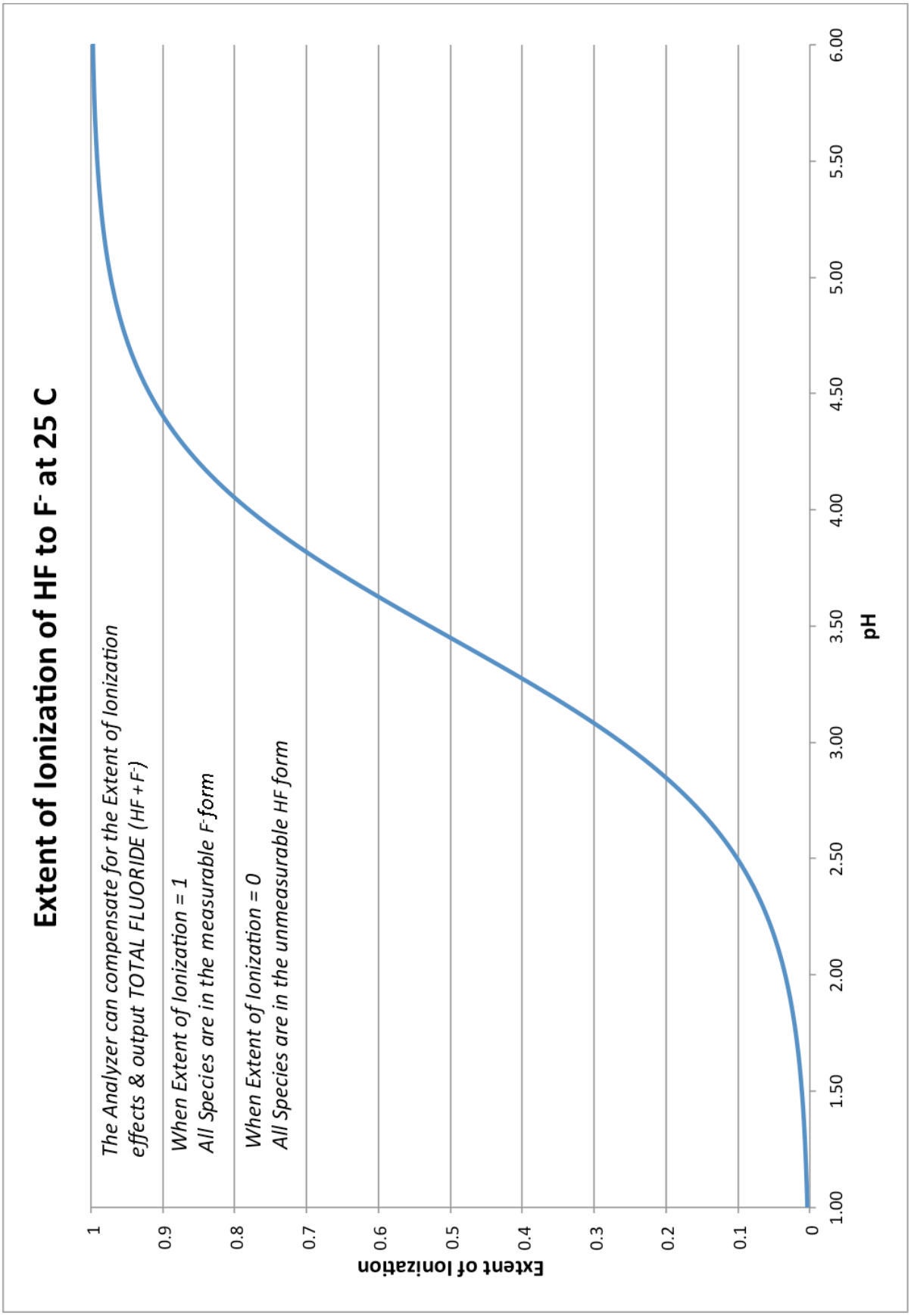

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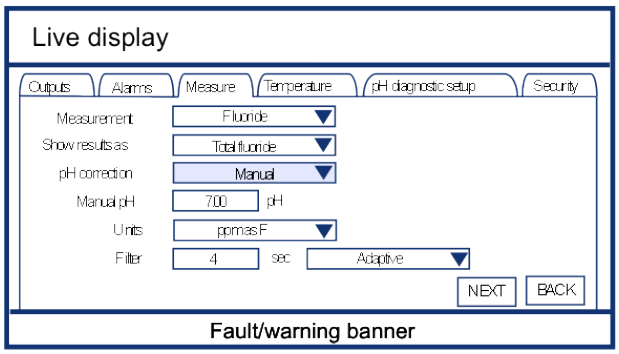
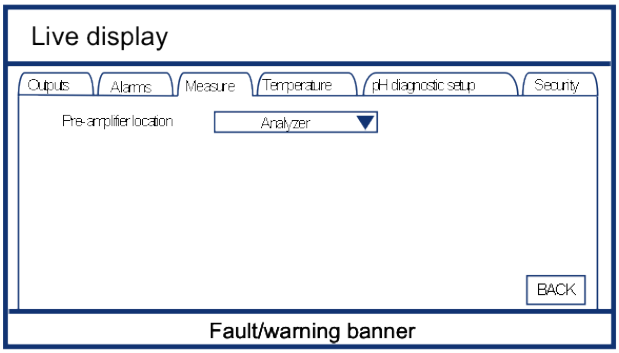
Advanced Configuration & Installation Guide for 56 Analyzers with pH Compensation Enabled for determination of "Total" unbound fluoride species

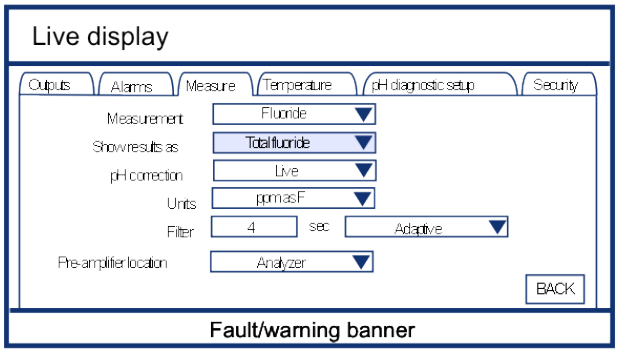
These steps are required for an installation of a new fluoride sensor in the advanced configuration:

1) Ensure Model 56 transmitter has the 2.19 or 2.30 software loaded before proceeding. Under the Measure Menu, select "Fluoride" for the channel 1 Measurement and "Total fluoride" as the selection for the Show result as field. You must 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 fluoride ISE sensor used without an integral preamplifier, which is the most typical for the AB 6100 style sensor with no more than 20 feet of cable. This setting can be toggled to the preamp in sensor choice when using an AB 6100 sensor that has an integral preamplifier to support long cable lengths, bridged lead terminations or else if an external preamplifier is employed.

2) Ensure that the fluoride ISE sensor is properly wired to the sensor channel 1 that you have configured for fluoride 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 fluoride 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 fluoride 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 or 2.30 special software will automatically correctly assume that the grab sample determined value enter for the "Standardize (grab)" calibration is the total unbound fluoride 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 fluoride 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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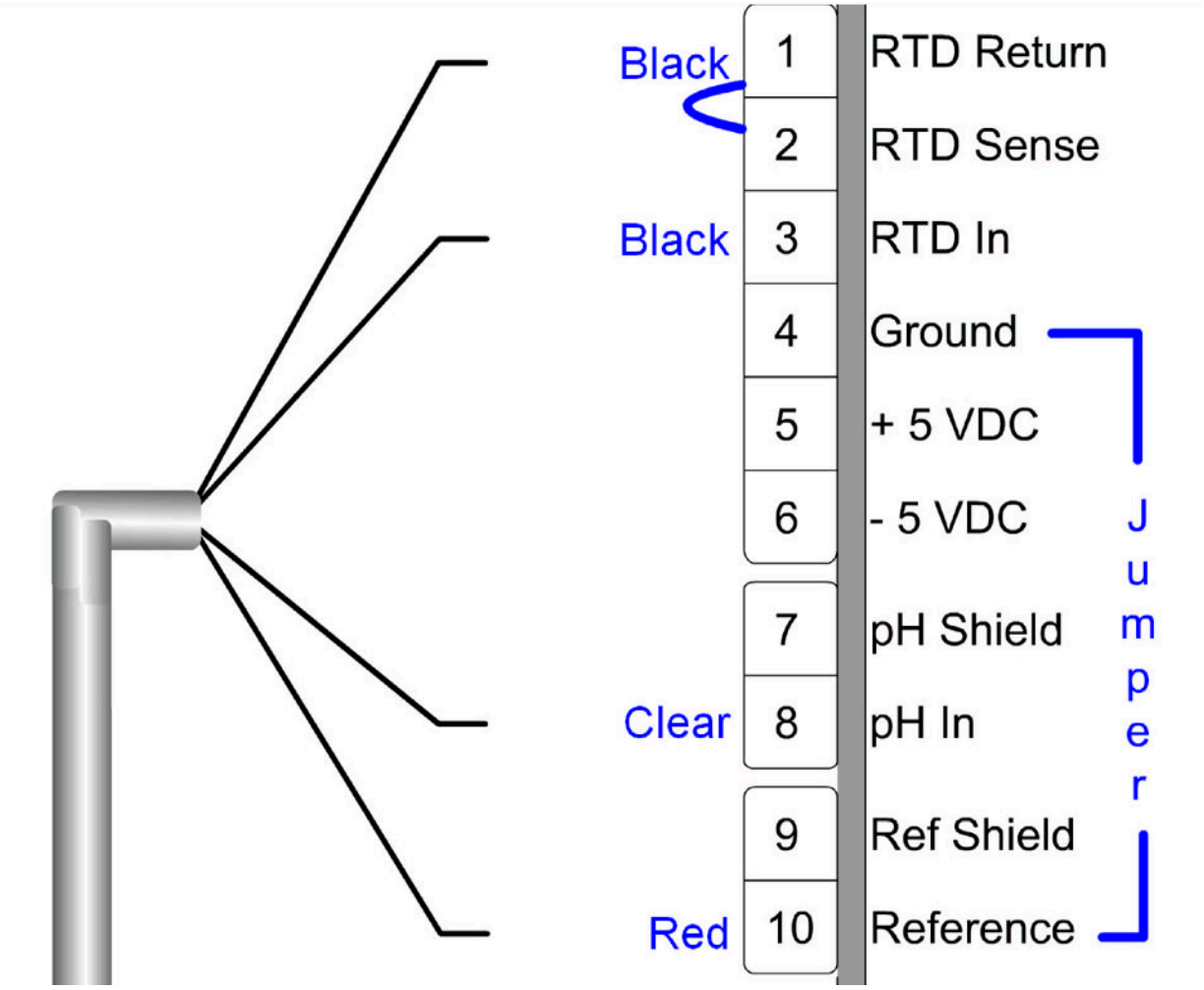
Connection Diagram of Iotron™ pH / ORP / ISE Sensors **Without** Preamplifiers to Rosemount 1056/1057/56 pH/ORP/ISE Analyzers

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 and Ion Selective Sensors, please put the active signal (clear) 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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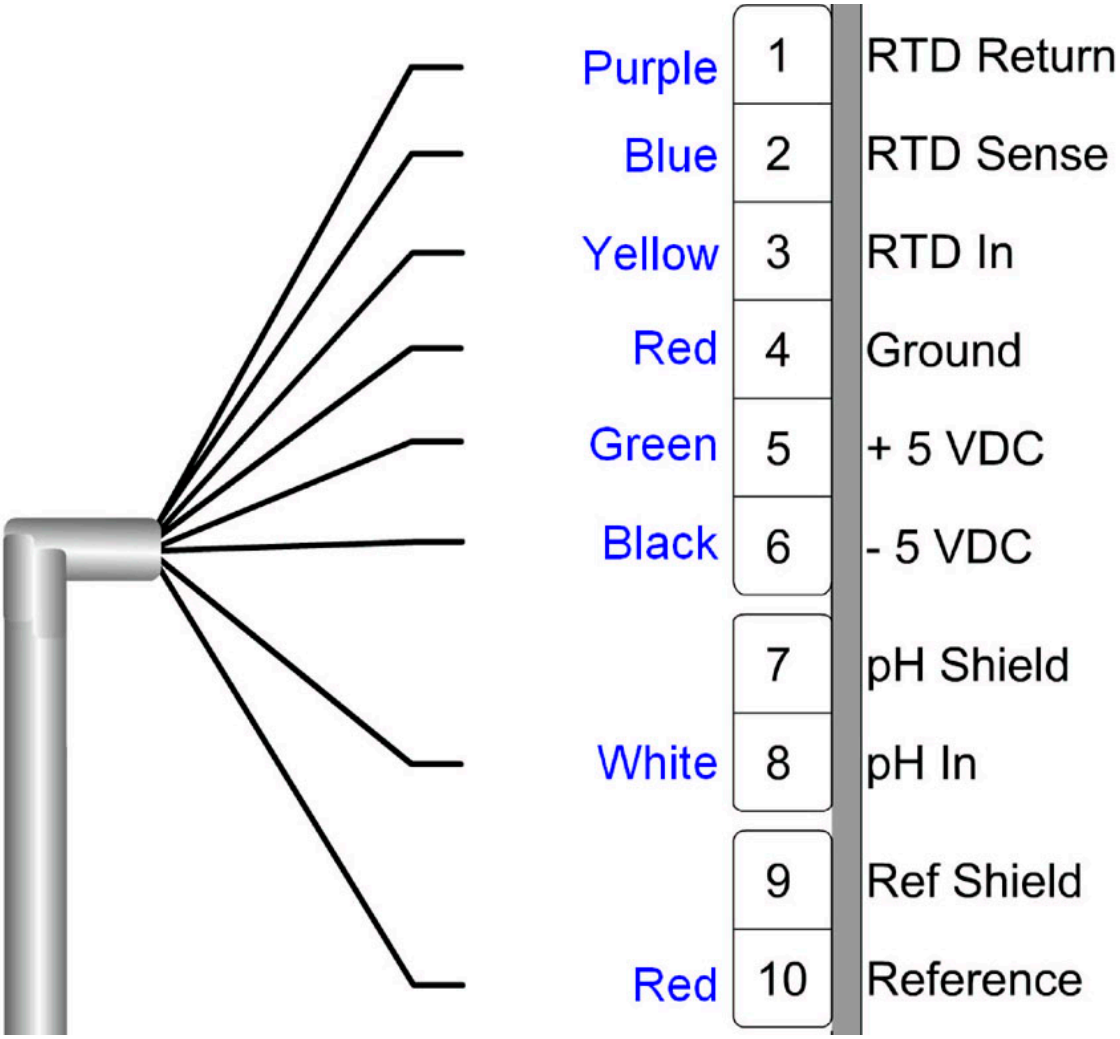
Connection from Iotron™ Sensor to Terminal Block in Rosemount Transmitter



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 fluoride 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 will need 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.

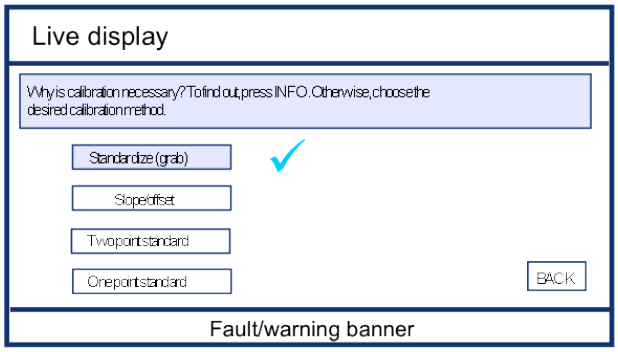
A grab sample should be taken from the process and analyzed by a suitable method for fluoride ion concentration. There are a variety of ways to perform the grab sample analysis including laboratory fluoride ion selective electrode methods adding the TISAB-II to the unknown sample to adjust for all background ion effects. When this method is used, it is typical to add the TISAB-II on a one to one volume basis to the unknown, and then to determine the fluoride concentration of this diluted sample. The actual fluoride concentration will then just simply be double the TISAB-II diluted unknown sample. Another method is to use a photometer to determine the fluoride concentration of the grab sample, typically available in ranges of 0-2ppm and 0-20ppm. This grab sample determined concentration of the process sample (by whatever method employed) will then 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:

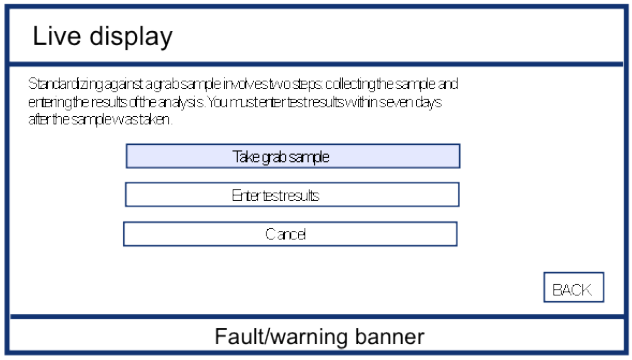
BOTH fluoride & pH sensors should be left in service the ENTIRE TIME this calibration is performed. Ensure that both sensors have been allowed sufficient time for stabilization before proceeding.

Steps for performing STANDARD (GRAB) Calibration

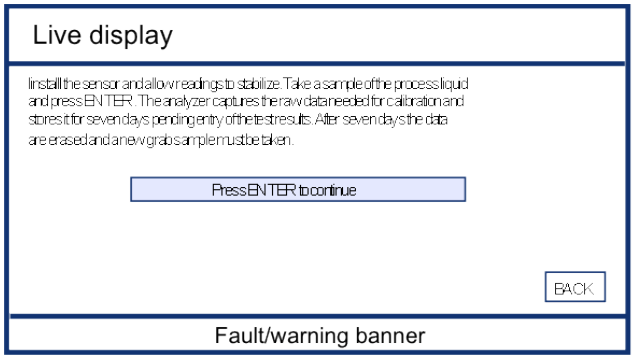
1. After choosing the sensor channel configured for fluoride measurement (Calibrate → S1 Measurement), you will be presented with the following calibration choices (see screenshot below):



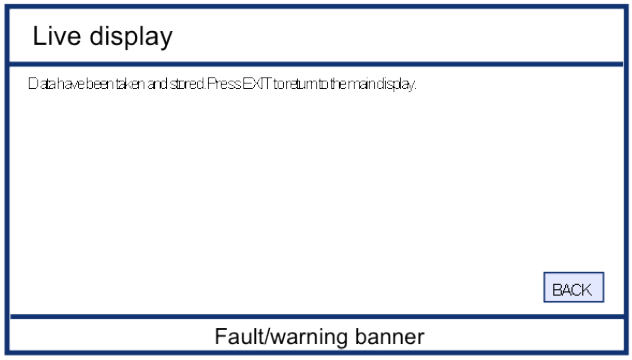
2. Once you have chosen the correct "Standardize (grab)" mode for the calibration you will be presented with the following next menu choices. Select the "Take grab sample" option:



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 or 2.30 software allows for a time delay up to seven days between when the grab sample is taken & 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.

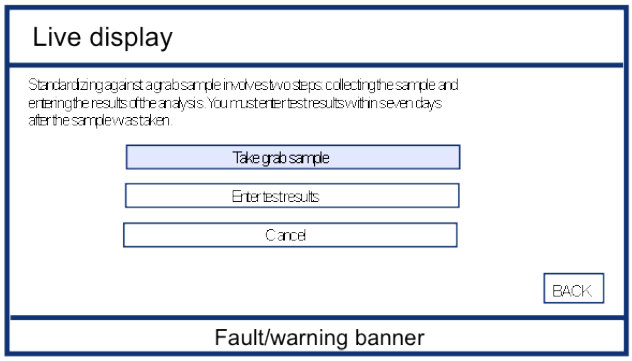


4. After pressing the "ENTER" key the software will store the time and date at which you took the grab sample for use with the onboard automatic correction for time induced changes to the inline readings.

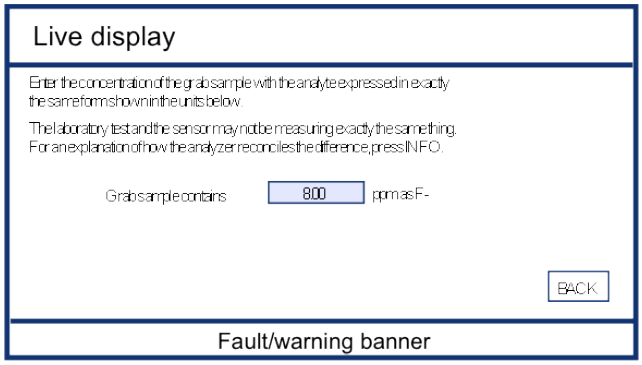


5. Analyze the grab sample taken by a suitable method in as timely a manner as possible. You will need to have this offline determined value to complete the standardize (grab) calibration process.

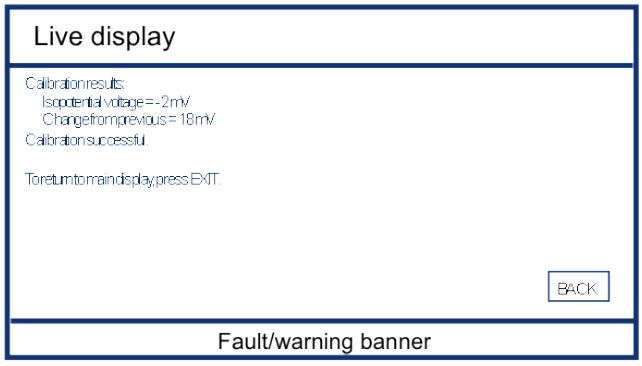
6. Navigate back to the menu options for the "Standardize (grab)", and choose the "Enter test results" option.



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 the Special

AB 6100 Fluoride Ion Selective (ISE) Type Sensors

Cleaning is generally only recommended if the tracking of the installed fluoride ISE sensor as compared to the periodic grab sample determinations diverges over the course of time. This might mean that some build-up has accumulated making it less responsive to the changes in fluoride 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.

SPECIAL NOTE & PRECAUTION:

The use of acid in the cleaning procedure is ONLY supported for the special AB 6100 type fluoride ISE sensors. The use of this acid cleaning regimen on any other sensor may render it completely 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. **If necessary, use 5% to 15% hydrochloric (HCl) acid to clean the fouling. Some common compounds that can tend to build-up for some wastewater treatment applications that are made more soluble in acid conditions include calcium fluoride (CaF_2) and fluorosilicates (various forms) amongst others.**
3. The fluoride sensor tip can be cleaned with isopropyl alcohol to remove any type of organic contaminants. No other solvents or reagents should be used without contacting ASTI to ensure that it is suitable.
4. 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.**
5. 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 solution after such a cleaning regimen before performing a subsequent standard (grab) calibration.
6. Any calibration fluoride 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 fluoride ion selective sensing crystal. The presence of even small air bubbles will cause erroneous readings and/or drift. See special notes below for resolution if you believe this issue is occurring for your sensor.
- The fluoride 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 and hook-up schematics found in the AB 6100 fluoride sensor shipping box.

Resolution for potential issue of air bubble on the AB 6100 fluoride sensor tip

Background:

There is a sealing washer cap on the AB 6100 style fluoride ion selective sensors. This allows for the long service life of these fluoride ISE sensor even with acid service in process and acid cleaning to remove potential fouling contaminants such as CaF_2 or various forms of fluorosilicates. The (unavoidable) trade-off is that the AB 6100 sensors are somewhat prone to getting an air bubble entrenched in this recessed area near the washer. If such an air bubble occurs the readings will be completely erratic. The three main solutions to this issue are:

1. Shakedown the sensor well in air. This usually liberates any internal air bubble entrenched as well as liberating any entrenched air bubbles that from on the outside of the sensor tip as well.
2. Shake the sensor in solution (calibration or process) at a 45-degree angle. This ensures that once immersed the bubble is liberated from the recessed cap show it have formed.
3. When installing into service make sure that the submersed sensor is installed at a 45-degree angle such that even if a bubble should form after it is placed into service it will not tend to persist.
4. Care should be taken to select an installation point that is the least prone for the formation of air bubbles form the process solution. Note that the AB 6100 type sensors can ONLY be installed as submersibles.

In the vast majority of cases following the procedures outlined above will resolve any air bubble induce anomaly to the fluoride ppm readings. If after following this procedure you still receive erratic readings please contact the factory for further assistance.

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2-Point Slope Calibration (OPTIONAL)

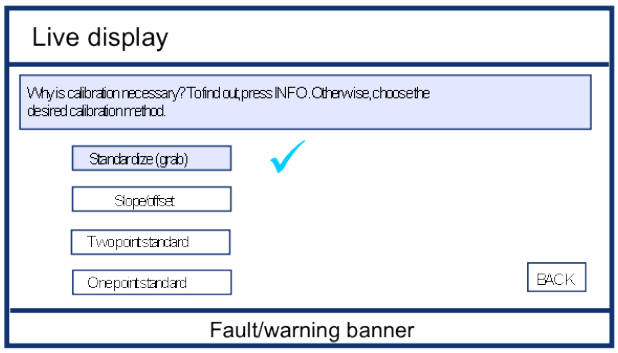
The slope response of the fluoride ion selective sensor (mV change per decade ion activity change) is a fundamentally characteristic value. The factory programmed slope value for fluoride ISE sensors rarely, if ever, needs to be adjusted. The vast majority of inline fluoride ion measurement applications 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 only induces only a drift in the absolute potential of the sensor, which is corrected by the standardize (grab) calibration. The aging of the sensor does not induce a change the sensor slope, even very near the end of service use (the slope stays constant over time).

IMPORTANT CAVEATS FOR A SUCCESSFUL 2-POINT SLOPE CALIBRATION:

- Fluoride ion calibration standards MUST have a TISAB-II type background. Procedures on how to fabricate your own fluoride ion standards with TISAB-II background are provided at the end of this ISE addendum. Alternatively, the following two suitable fluoride ion calibration standards can be purchased from any major commercial chemical supplier.
    - Fluoride 1 ppm as F- with TISAB II, Orion # 040906
    - Fluoride 10 ppm as F- with TISAB II, Orion # 040908
- 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 1ppm standard before starting your 2-point slope calibration procedure.
- Use the low 1ppm standard for the first calibration point, and then use the high 10ppm standard for the second calibration point. Allow sufficient time for the reading to stabilize before proceeding to calibration to the high 10ppm 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 fluoride 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 fluoride ion calibration standard (1ppm if you purchased the Orion # 040906) followed by entering the value for your second high fluoride ion calibration standard (10ppm if you purchased the Orion # 040908). After completion of the 2-point slope calibration the slope obtained from the procedure will be reported.

The factory default value is -57.16mV per decade for the fluoride ion selective sensor slope. If you obtain a slightly different value with your 2-point slope calibration, this is most likely due to degradation of the standard itself or else some minor suboptimal part of how the calibration procedure was performed. 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 changed 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 FLUORIDE 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 Fluoride Standard Solutions

Materials

Sodium Fluoride (Analytical or ACS Reagent Grade or better, brand new sealed dry bottle preferred)
- *Spectrum Catalog # S1280*
TISABII (Total Ionic Strength Adjustor Buffer)
- *Spectrium Catalog # 246-25171, Orion # 940909*
1 Liter Volumetric Flask (one each minimum, four each recommended)
5 Liter Volumetric Flask (one each) or 2 Liter Volumetric Flask (two each)
1 mL volumetric pippete
10 mL volumetric pippete
1 liter opaque plastic bottles with air-tight sealing cap (five each)
DI Water (15 MegaOhms or higher resistivity grade recommended but not critical)

- ENSURE THAT ALL GLASSWARE IS CLEAN AND DRY BEFORE PROCEEDING.
- THOROUGHLY CLEAN EACH VOLUMETRIC FLASKS AFTER PREPARING ANY SOLUTION WITH DI WATER.
- SOLUTION PREPARED FROM THIS PROCEDURE WILL STAY GOOD FOR 1 YEAR FROM DATE OF MANUFACTURE IF STORED IN AN SEALED, OPAQUE PLASTIC BOTTLE IN COOL DRY LOCATION

Stock Solution Preparation Procedures:

Preparation of Diluted TISAB II background stock solution (DO THIS FIRST!):

1. Measure out 2 liters of TISAB II solution and pour into a 5 liter volumetric flask.
2. Dilute with DI water to 5 liter mark. Mix solution well until all the two solutions are completely miscible and the resulting solution is homogeneous.
3. Seal 5 liter volumetric flask with glass stopper.

Preparation of 10,000 ppm Fluoride stock solution (DO THIS FIRST!):

1. Measure out 22.101 grams of sodium fluoride salt.
2. Place this sodium fluoride into 1 liter volumetric flask.
3. Dilute with DI water to the 1 liter mark. Mix solution well until it is completely homogeneous (dissolved).
4. Transfer this 10,000 ppm fluoride stock solution to a 1 liter plastic bottle and label appropriately.

Fluoride Calibration Solution Preparation Procedures:

Preparation of 10 ppm Sodium Fluoride Standard Ion Solution

1. Draw 1 mL of 10,000 ppm fluoride stock solution and transfer to a 1 liter volumetric flask.
2. Dilute with TISAB II diluted background solution to 1 liter mark. Mix solution well until completely homogeneous.
3. Transfer this 10 ppm fluoride calibration solution to a 1 liter plastic bottle and label appropriately.

Preparation of 100 ppm Sodium Fluoride Standard Ion Solution

4. Draw 10 mL of 10,000 ppm fluoride stock solution and transfer to a 1 liter volumetric flask.
5. Dilute with TISAB II diluted background solution to 1 liter mark. Mix solution well until completely homogeneous.
6. Transfer this 100 ppm fluoride calibration solution to a 1 liter plastic bottle and label appropriately.

Preparation of 20 ppm Sodium Fluoride Standard Ion Solution

7. Draw 2 mL of 10,000 ppm fluoride stock solution and transfer to a 1 liter volumetric flask.
8. Dilute with TISAB II diluted background solution to 1 liter mark. Mix solution well until completely homogeneous.
9. Transfer this 20 ppm fluoride calibration solution to a 1 liter plastic bottle and label appropriately.

Preparation of 200 ppm Sodium Fluoride Standard Ion Solution

10. Draw 20 mL of 10,000 ppm fluoride stock solution and transfer to a 1 liter volumetric flask.
11. Dilute with TISAB II diluted background solution to 1 liter mark. Mix solution well until completely homogeneous.
12. Transfer this 200 ppm fluoride calibration solution to a 1 liter plastic bottle and label appropriately.

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