IMPORTANT NOTES FOR FLUORIDE ISE

MEASUREMENT SYSTEM WITH 56 ANALYZER

IMPORTANT NOTE # 1:

This addendum <u>ONLY</u> 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. <u>IMPORTANT NOTE # 2:</u>

This addendum <u>ONLY</u> 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 fluoride configuration. Specifically, the pH compensation is NOT enabled in the described use. For applications where the pH will be 5.5 or lower, please refer to the advanced configuration with pH compensation enabled. IMPORTANT NOTE # 4:

There <u>MUST</u> 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.

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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 suitable temperature range of the AB 6100H or AB 8100 fluoride ISE sensors is five to fifty (5-50) degrees Celsius (41 to 122 degrees Fahrenheit). The supported pH range of the AB 6100H or AB 8100 fluoride ISE sensors is 5.5 to 9.5. A pH greater than 5.5 eliminates the need for compensation of the pH effects on fluoride concentration and for optimum sensor lifetime and a pH of less than 9.5 to minimizes aging on the measuring crystal itself.

If the pH will consistently operate below 5.5, use the AB 6100 fluoride ion selective sensor designed for such process conditions, NOT the AB 6100H or AB 8100 fluoride ion selective sensors. Please note that the AB 6100 fluoride ion selective sensor for low pH applications is <u>NOT</u> suitable for low-flow panel or inline installations. The AB 6100 type sensors should ONLY be used for immersion/submersion installation schemes. The AB 6100 style sensor can continuously support a pH range from 0 to 9.5 (intermittently 11) and temperatures from 5 to 70 degrees Celsius (41 to 122 degrees Fahrenheit).

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 configuration of the dual channel 56 analyzer such that calibration, display and output will be in the total pH compensated "Total" unbound fluoride units (see link below for a more detailed discussion):

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

Please contact the factory for applications where the pH will be below 5.5 for further assistance.

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Basic Configuration & Installation for 56 Analyzers for Free Fluoride Only (without pH Compensation Enabled for "Total" unbound fluoride species)

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

 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 "Fluoride" choice should be selected for the channel to which the fluoride ISE sensor will be connected (see screenshot below as a guide). This screenshot below shows the sensor without an integral preamplifier, which is the typical for the AB 8100 style sensors, supplied for most inline installations, although this can be toggled to the preamp in sensor choice when appropriate.

Live display			
Outputs Alarms Meas	sure Temperature	pH diagnostic setup	Security
Measurement	Fluoride		
Show results as	Freefluoride		
Units	ppmas F		
Filter	4 980	Adaptive	▼ *sæ(1).
Pre-amplifier location	Analyzer		
		\checkmark	
			BACK
Fault/warning banner			

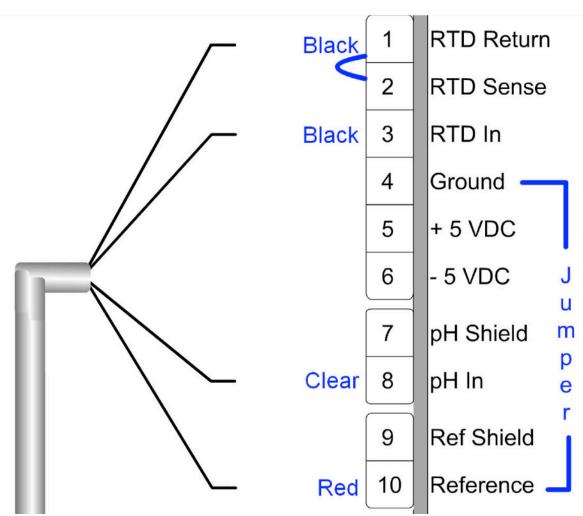
 Ensure that the fluoride ISE sensor is properly wired to the sensor channel that you have configured for fluoride ion measurement. Find below links to the wiring detail both for sensors with and without integral preamplifiers:

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 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.

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



Connection from IotronTM 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).

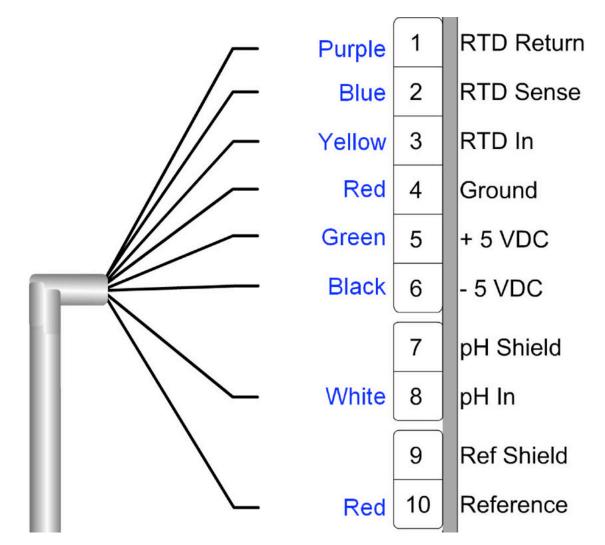
Note 3: Terminals 4 & 10 and terminals 1 & 2 must be tied together to satisfy the analyzer input requirements and disable the reference diagnostic features (pH glass diagnostics should still be available).

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

Connection from IotronTM 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:

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 fluoride measurement (Calibrate \rightarrow S1/S2 Measurement), you will be presented with the following calibration choices (see screenshot below):

Live display	
Why is calibration necessary? Tofind out, press INFO. Otherwise, choose the desired calibration method.	
Standardize (grab)	
Slopeloffset	
Oreportstandard	3ACK
Fault/warning banner	

603 North Poplar Street Orange CA 92868-1011 USA Web Site: http://www.astisensor.com/cgi-bin/ttx.cgi

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:

Live display		
	inst agrabsample involves two steps: collecting the sample and softre analysis. You must enter test results within seven days astaken.	
	Take grab sample	
	Ententestresults	
	Cancel	
		BACK
Fault/warning banner		

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.

Live disp	blay	
and press BN TBR storesit for seven d	ndallowreadingsto stabilize. Take a sample of the processilopud ?. The analyzer captures the raw data needed for calibration and ays pendingentry of the testnesuts. After seven days the data awgrab sample must be taken. Press BNTER to continue	
		BACK
	Fault/warning banner	

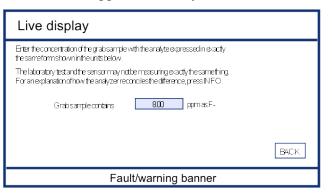
4. After pressing the "ENTER" key the software will store the time and date at which you took the grab sample for use with the onboard automatic correction for time induced changes to the inline readings.

Live display	
Data have been taken and stored. Press EXIT to return to the main display.	
	BACK
Fault/warning banner	

- 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.

Live dis	play	
	inst agrabsample involvestivosteps collecting the sample and softre analysis. You must entertestresults within seven days astaken.	
	Take grab sample	
	Ertertestresults	
	Cancel	
		BACK
	Fault/warning banner	

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)

Live display	
Calbrationresults Isopatential voltage =- 2mi/ Changefromprevious = 18mi/ Calibration successful. Toretum tormain display, press ExIT.	
	BACK
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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<u>Cleaning and Maintenance of</u> <u>AB 6100H or AB 8100 Fluoride ISE Sensors</u>

Cleaning is generally only recommended if the tracking of the inline fluoride 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 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.

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 fluoride 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 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.
- 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 6100H or AB 8100fluoride sensor shipping box. This data is included at the end of this ISE addendum for convenience as well in case the hard copy originally provided was lost or misplaced.

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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 <u>MUST</u> 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 \rightarrow 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)		
Slopetifiset		
Twopcintstandard		
Oreportstandard	BACK	
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 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 <u>MUST</u> 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*Liter Volumetric Flask (one each minimum, four each recommended)
Liter Volumetric Flask (one each) or 2 Liter Volumetric Flask (two each)
mL volumetric pippete
mL volumetric pippete
titer 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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