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IMPORTANT NOTES FOR FLUORIDE ISE SYSTEM WITH 1056/1057 ANALYZER

Calibration and Cleaning of Fluoride Selective Ion Measurement System For Water and near Wastewater Fluoride Analysis

Before proceeding further, it is recommended that a review of the following technical documents that describes the general provisions for online ion selective measurements:

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

All calibration solutions and process grab sample should be calibrated and tested at identical temperatures to the process temperature for optimal results. The actual temperature of the process solution (and thereby the calibrating solutions as well) is not as critical as the fact that they are calibrated at the same temperature to eliminate all potential sources of uncertainty. For greatest overall accuracy of the fluoride (F^-) measurement, however, all tests should be performed as close to 25 degrees Celsius (room temperature process solutions) as possible. The valid (permissible) temperature range for all fluoride ion selective sensors is five to fifty (5-50) degrees Celsius (41 to 122 degrees Fahrenheit).

Calibration Point 1 (always the lower concentration value) and Calibration Point 2 (always the higher concentration value) determines the response curve of a given fluoride Ion Selective Sensor (AB 8100 or AB 6100H). The calibration value for point 1 is the low ppm solution and point 2 is high ppm solution. The one point offset calibration using a process grab sample is always performed by using the standardize option from the calibrate main menu. A one point calibration should never be performed by using the 2-point calibration option from the calibrate Main Menu.

The calibration solutions should be kept clean and out of direct sunlight and/or other high-energy radiation sources to maximize accuracy of their ppm values. New sensors should be conditioned in fluoride standard solution for 3 - 5 minutes before beginning the calibration procedure as described in this guide.

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The recommended pH range of the AB 8100 and AB 6100H sensors is 5.5 to 9.5 on a continuous basis, although measurement down to a pH of 3 and up to a pH of 11 on an intermittent basis is possible. For continuous long-term use, ASTI recommends a pH greater than 5.5 to eliminate the need for compensation of the pH effects on fluoride concentration and for optimum sensor lifetime and less than 9.5 to minimize aging on the cyrstal. **If the pH will consistently operate below 5.5, use the AB 6100 fluoride ion selective sensor designed for such conditions, NOT the AB 6100H or AB 8100 sensors. Please note that the AB 6100 fluoride ion selective sensor for low pH applications is NOT suitable for low-flow planel installations and may have some slight calibration issues as it is designed to operate and measure in a low pH environment.**

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. To convert the measured fluoride ion activity (free fluoride) into total fluoride the free fluoride measurement must be compensated for pH. See the pH dependent extent of ionization curve for HF dissolved gas and fluoride ions (F^-) for a graphical representation of this phenomenon. Compensation for the effect of pH on the extent of ionization for ammonia may be required for pH values below 5.5 to provide complete total fluoride data. Please also note that these pH effects is a temperature dependent phenomenon. The provided extent of ionization curve is only completely valid for pure two component systems with deionzied (DI) water. Real world water solutions of a much more complex makeup may vary somewhat from these idealized curves, although the deviation is not expected to be vast for most typical systems. The algorithm to compensate for these pH dependent extent of ionization of HF to fluoride ions (F^-) is available upon acceptance of a simple licensing agreement from ASTI. For further details please contact the ASTI factory about requesting this algorithm.

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

1) Enter the nominal ISO Voltage -47 mV and Slope (- 57.16 mV per decade)
2) Perform a two point calibration to empirically determine slope (see procedure on following pages).
3) Place sensor into process and allow it to find electrochemical and thermal equilibrium. The time required for this may vary depending upon the particular application.
4) To account for any differences between the ionic strength and interfering ions between calibration and measured solution, a grab sample should be taken and analyzed by a suitable analysis system, and the online fluoride ion selective measurement system adjusted to read the grab sample analyzed value. The sensor should be left continuously in service and this grab sample offset calibration performed as may be required, unless the sensor seems to be losing sensitivity, giving erratic readings or requires cleaning. The 1-point offset calibration is called “Standardize” in the Rosemount ISE analyzer calibrate sub-menu.

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Configuring 1056 Analyzers for Fluoride Measurements on Channel 2 of Low Flow FCL Panels

For any systems where there is a single channel ISE configuration or a dual channel ISE/pH configuration the ISE sensor will always be configured to the channel 1. On a 1056 channel 1 is on the middle position and for a 1057 it is on far right position. You should check that your ASTI ISE sensor is properly wired according to the official ASTI hook-up schematic for ASTI sensors without preamplifiers to the 1056 analyzer (also provided later in this guide for convenience). The hook-up schematics are identical for the 1056 and 1057 although the board positions are different.

http://www.astisensor.com/Rosemount_1056_No_Preamp_Hookup.pdf
http://www.astisensor.com/Rosemount_1056_With_Preamp_Hookup.pdf

The 1056 analyzers will support both ASTI ISE sensors with and without preamplifiers, although preamplifier are generally not required for cable length of less than 20 feet. The ISE channel MUST ALWAYS be set to Custom ISE (If your analyzer shows the measurement anything OTHER THAN Custom ISE IT IS IMPROPERLY CONFIGURED). If your analyzer has a setting of anything other than Custom ISE, it has undoubtedly been (accidentally) improperly reconfigured after the ASTI factory configuration and testing. Here is how to restore it:

Main Menu -> Program -> Measurement -> Sensor 1 -> Custom ISE Setup ->
{{ENTER SLOPE, FORM. WT., ISO PCON, & ISO VOLTAGE}}

The values for these four Custom ISE variables are provided on the following pages along with an explanation of their meanings. Normally you should not need to enter these values at all as all ASTI provided 1056 ISE analyzer are preconfigured and tested with the ISE sensor provided at the factory. Only an alteration of the factory configuration would necessitate a restoration of ASTI factory configuration.

NOTE:: THE RESETTING OF THE ISE CONFIGURATION AS DESCRIBED ON THE FOLLOWING PAGE **IS NOT** THE SAME AS “RESTORE TO ROSEMOUNT FACTORY DEFAULT”. IN FACT RESTORE TO ROSEMOUNT FACTORY DEFAULT WILL ENSURE THAT ALL RELEVANT SETTINGS ARE COMPLETELY LOST (YOU WILL NEED TO START FROM SCRATCH IF YOU ACCIDENTALLY INVOKE THIS OPTION).

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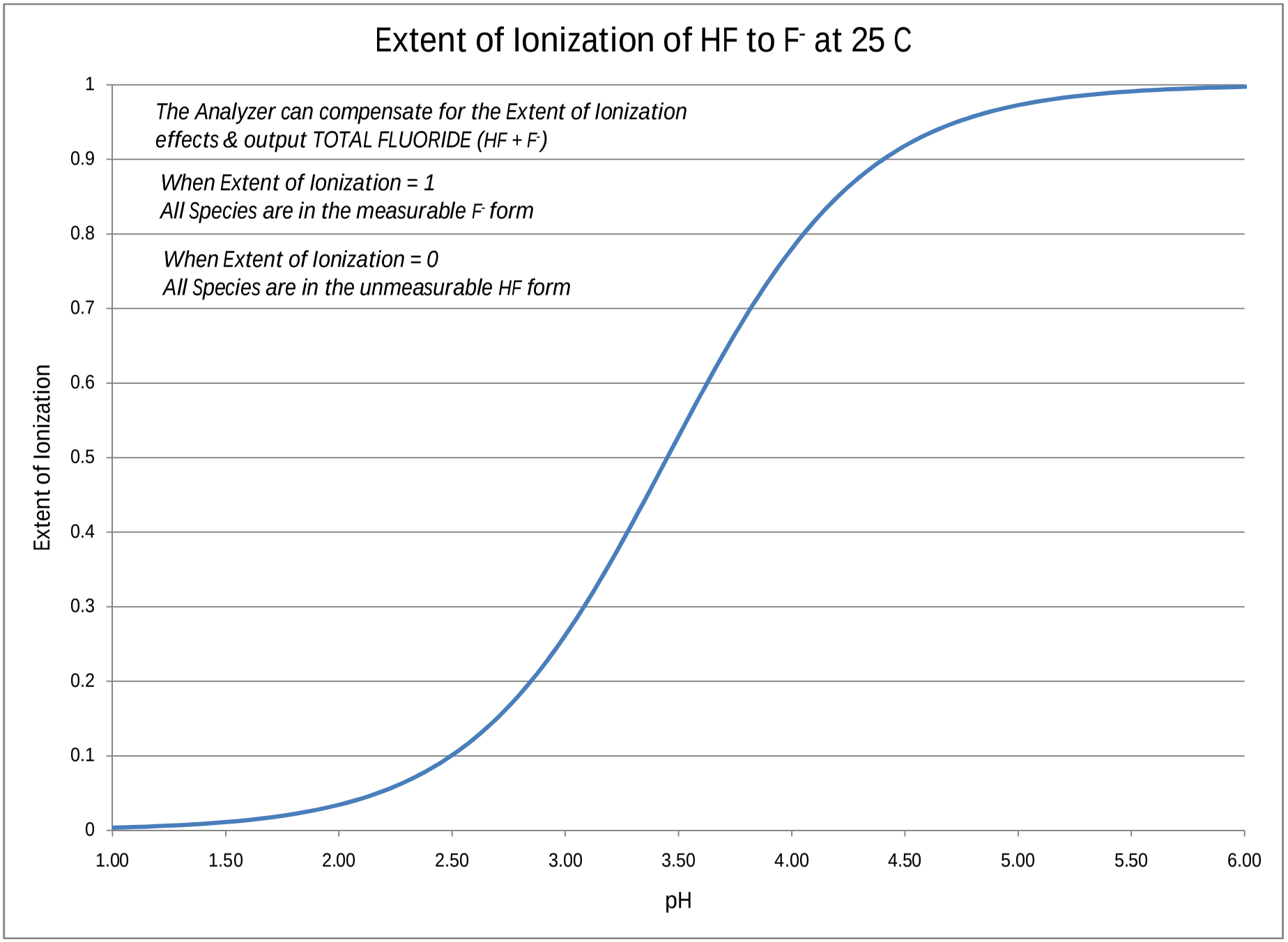
Custom ISE Variable	**Description of Variable**	**NOTES**
19.00 grams per mol	IONIC WEIGHT **Form Wt.** in the 1056 analyzer terminology	Defined by Selective Ion Measurement – DO NOT MODIFY
2.13263	ISOPOTENTIAL CONCENTRATION **Iso pCon** in the 1056 analyzer terminology	Factory Defined – DO NOT MODIFY
-57.16 mV per decade	DEFAULT SLOPE **Slope** in the 1056 terminology	Will be changed when 2-point calibration is performed
-47 mV	ISOPOTENTIAL VOLTAGE **Iso Voltage** in the 1056 analyzer terminology	Will be changed when either a 2-point slope or 1-point offset standardize calibration is performed

Please note that after restoring the analyzer to the proper Custom ISE configuration (as described above), you will need to repeat your 2-point calibration using calibration solutions that are one decade (10X) apart in value. In addition, you will need to once again place the ISE sensor back into service and allow it to equilibrate. You will then also need to repeat your 1-point grab sample calibration (standardize in the 1056 terminology). This means taking a sample from the process and determining the ISE concentration (in ppm) for the grab sample and then using the 1-point standardize calibration feature of the 1056 analyzer to bring the online reading in accordance with the laboratory grab sample determination.

In addition, once you reset your analyzer with the correct Custom ISE configuration, you can choose to run the temperature compensation in the automatic mode (this is the default) or in the manual temperature compensation mode. Control of the temperature compensation settings are identical for the Custom ISE and pH channel, namely in this particular case:

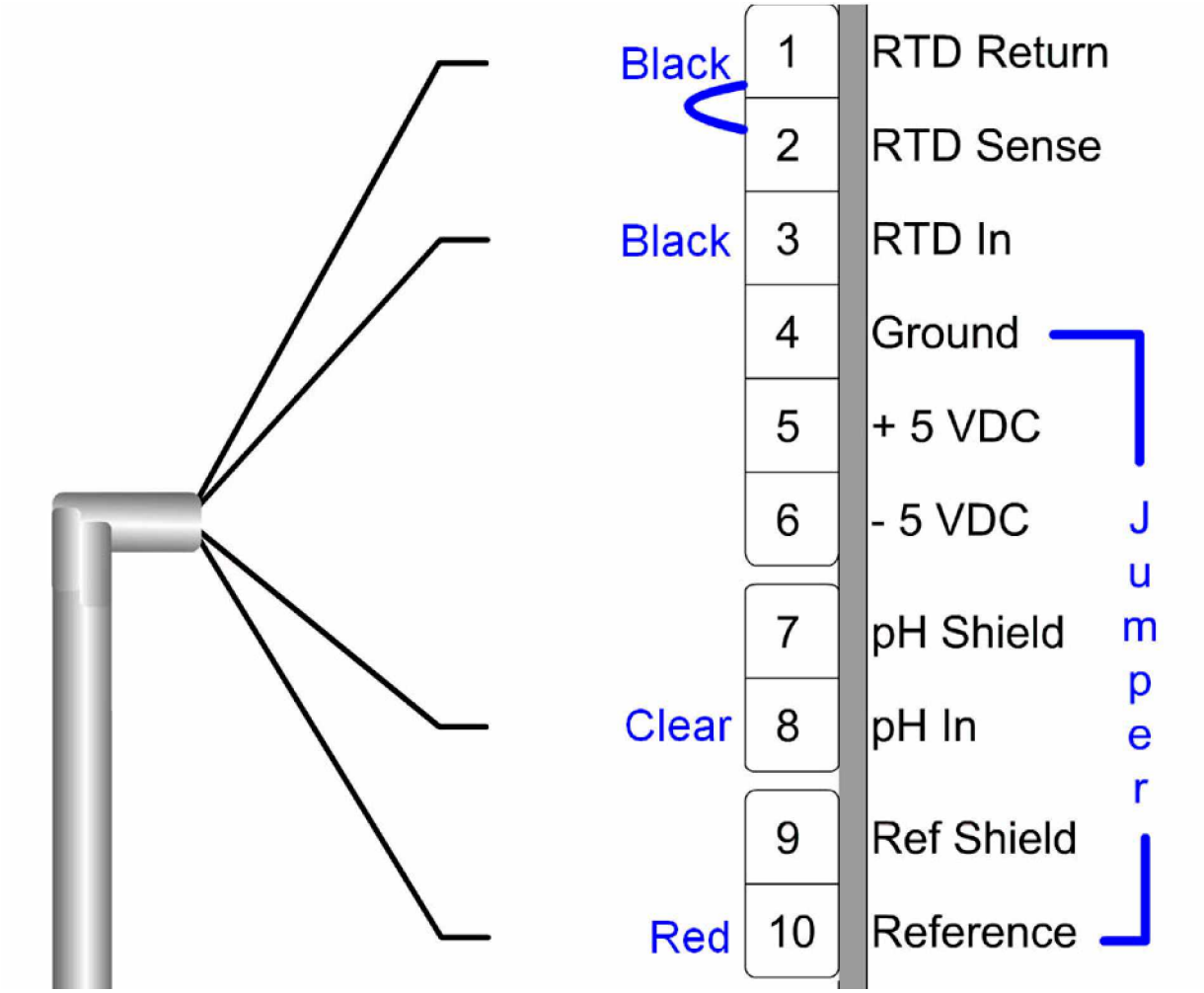
Main Menu -> Temperature -> Sensor1 -> {{ Set for AUTO or MANUAL }}

Please also note that the activity of free fluoride ions in solutions is pH and temperature dependent over some pH and temperature ranges. The extent of ionization (HF) conversion to the measurable (F^-) ion form is a pH and temperature dependent process. The following page describes such dependence. Contact the factory for additional assistance with this technical issue.


Extent of Ionization of HF to F- at 25 C
The Analyzer can compensate for the Extent of Ionization effects & output TOTAL FLUORIDE (HF + F-)
When Extent of Ionization = 1
All Species are in the measurable F- form
When Extent of Ionization = 0
All Species are in the unmeasurable HF form
Extent of Ionization
1
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
1.00
1.50
2.00
2.50
3.00
3.50
4.00
4.50
5.00
5.50
6.00
pH

Connection Diagram of Iotron™ pH / ORP / ISE Sensors **Without** Preamplifiers to Rosemount 1056 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).

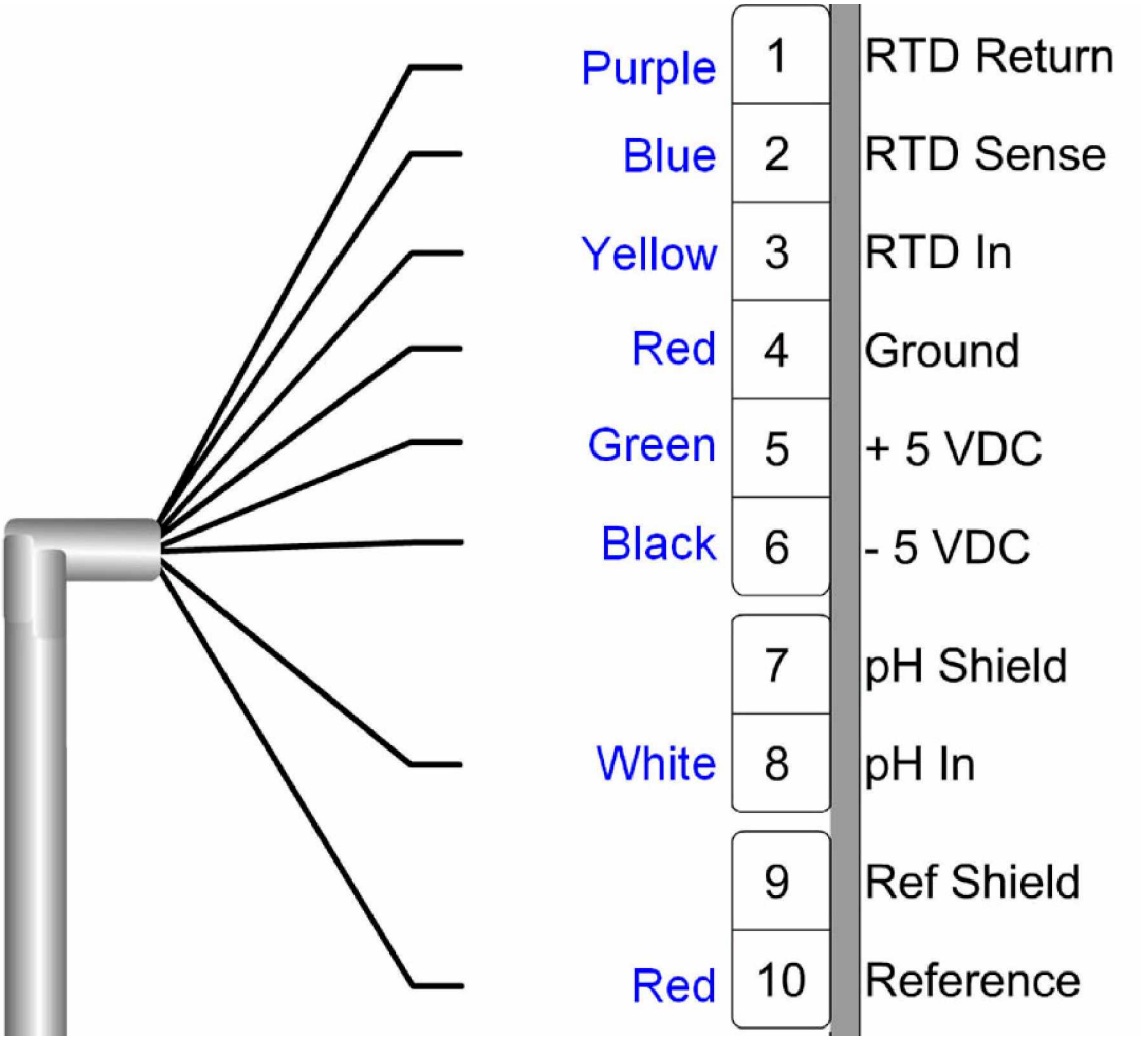
Note 2: For ORP and Ion Selective Sensors, please put the active signal (clear) to terminal 8 (indicated as pH In).

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 Channel Analyzers, please ensure that the proper type of sensor is connected to the proper input board.

Connection Diagram of Iotron™ pH / ORP / ISE Sensors **With** Preamplifiers to Rosemount 1056 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 Channel Analyzers, please ensure that the proper type of sensor is connected to the proper input board.

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Fluoride (F^-) Probe Two Point Calibration

This calibration method should not need to be performed frequently. All new fluoride ion selective sensors should be undergo a 2-point calibration first. This two-point calibration determines the sensitivity or slope of each sensor, which is then stored in the analyzer. Subsequent 1-point offset calibrations can be made using only the standardize option from the calibrate main menu. A "standardize" (a.k.a. 1-point) offset calibration must be performed after every 2-point calibration. Details for the 1-point calibration are given in the proceeding page. **MAKE SURE THAT THE "HOLD" FEATURE IS ON BEFORE STARTING ANY CALIBRATION. READ THE ROSEMOUNT 1056 MAIN MANUAL FOR FURTHER DETAILS ABOUT THE HOLD FEATURE.**

Set-up requirements:

Two 250 mL GLASS OR PLASTIC BEAKERS

(Preferably heavy enough so that the ISE sensor does not tip over the beaker!)

Low Fluoride Standard Solution (low ppm)

High Fluoride Standard Solution (high ppm)

Follow the on-screen directions in 2-point calibration submenu in the 1056 Analyzer. The low standard solution (low ppm) will always be Calibration Point 1 & the high standard solution (high ppm) will always be Calibration Point 2. The exact values for these low and high fluoride standard solutions will depend upon your exact needs. Very many fluoride calibration solutions are commercially available from a variety of chemical supply houses. **Be sure before purchasing any such commercial calibration solutions that they have a TISAB-II background and that they values will bracket you desired measurement range.**

Alternatively, instructions on how to prepare your own fluoride calibration standards using the typical in-house ASTI fabrication procedure can be obtained by using the URL link below.

http://www.astisensor.com/Calibration/Fluoride_Calibration_Solutions_Preparation_Procedure_06-02-05.pdf

These procedures are also included in this addendum for convenience. Either the commercially available fluoride calibration standards or ASTI procedure prepared standards will allow you to successfully perform a two-point slope calibration.

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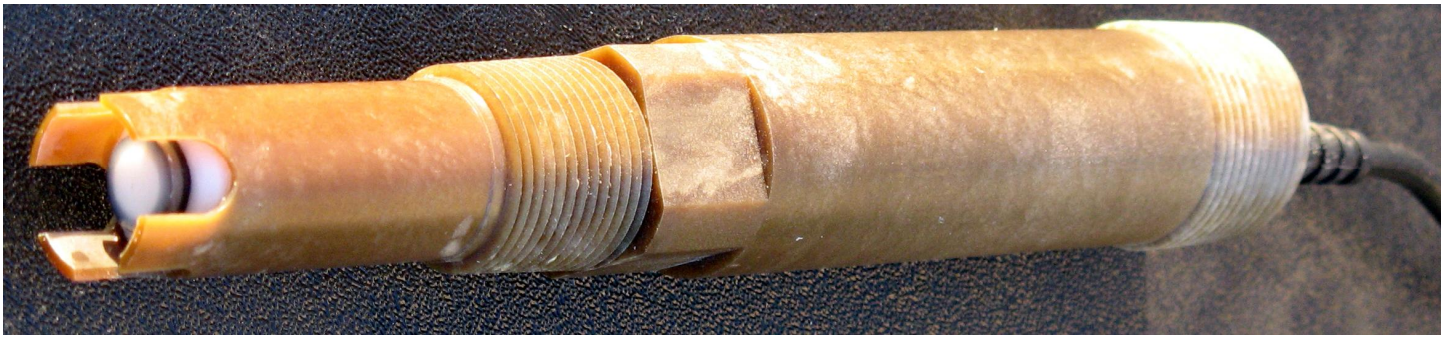
Important Notes about Calibration:

- Fill a 250 mL GLASS beaker with enough standardization solution such that the entire tip of the Fluoride sensor will be submersed
- Please read sheet on the cleaning procedure for this ion selective sensor. The cleaning procedure should usually be performed when transferring a sensor in or out of solution. Sensors can also be cleaned before being placed into different concentration standardization solutions and/or grab sample solutions. Cleaning is only required if fouling appears on the ISE membrane or on the reference junction. If no contamination is apparent, then the simple rinsing procedure can be used (see following sections for details).
- Thoroughly rinse the sensors with DI water and gently blot dry with a clean paper towel. Be careful not to scratch or damage the sensitive fluoride ion selective membrane
- Allow a minimum of 3 – 5 minutes for the sensor to stabilize once it has been removed from the process and placed into the low standard solution (low ppm).
- Allow a minimum of 3 – 5 minutes for the sensor to stabilize between the low and high Calibration Solutions (low ppm to high ppm).

Key Sequence for 2-Point Calibration

1. Press the MENU button
2. Select Calibrate. Press ENTER.
3. Select Sensor 1 or Sensor 2 corresponding to Custom ISE. Press ENTER.
4. Select 2-point Calibration. Press ENTER.
5. Fluoride sensor should be in low ppm standard solution already cleaned and conditioned. Press Continue. Instrument will display that Calibration Point 1 is stabilizing. The instrument will take about 20 seconds to stabilize and determine mV value for the first calibration point.
6. After Cal Point 1 has stabilized select edit and input low ppm into instrument**. This value must be entered even if it already is correct on the display**. After low ppm has been entered into analyzer, press save to continue to second calibration point.
7. Fluoride sensor should be in high ppm standard solution already cleaned and conditioned. Press Continue. Instrument will display that Calibration Point 2 is stabilizing. The instrument will take about 20 seconds to stabilize and determine mV value for the second calibration point.
8. After Cal Point 2 has stabilized select edit and input high ppm into instrument. **This value must be entered even if it already is correct on the display.** After high ppm has been entered into analyzer, press save.

THE ROSEMOUNT ANALYZER AT THIS POINT SHOULD SNAP TO THE NOMINAL PPM VALUE OF THE HIGH FLUORIDE PPM STANDARD SOLUTION. IF THIS DOES NOT OCCUR, REPEAT STEPS AS OUTLINED ABOVE UNTIL THE ANALYZER ACCEPTS THE TWO-POINT CALIBRATION. YOU CAN ALSO CHECK THE SLOPE VALUE UNDER DIAGNOSTIC VARIABLES TO ENSURE THAT IT SHOWS SOMETHING REASONABLE.



The ion selective sensor shown above is a representative picture for visualization purposes. Your particular sensor may appear somewhat different to that shown above for a variety of reasons.

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Single Point (1-Point) Offset Calibration – Grab Sample

This is the correct method to Adjust for Sensor Drift!!

Fluoride Sensor One Point Calibration ("Standardize")

Only use "Standardize" function in Calibrate Menu.

Can be performed as frequently as may be required**.**

When the fluoride sensor has been calibrated by the 2-point method previously described only a "single point" grab sample offset calibration should be required thereafter. **MAKE SURE THAT THE "HOLD" FEATURE IS ON BEFORE STARTING ANY CALIBRATION. READ THE ROSEMOUNT 54E-PH/ORP MANUAL FOR FURTHER DETAILS ABOUT THE HOLD FEATURE**.

Set-up requirements:

Two 250 mL GLASS OR PLASTIC BEAKERS

Process Grab Sample Solution

A grab sample should be taken from the process and analyzed by an alternate 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 bassis 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. This grab sample determined concentration of the process sample (by whatever method employed) will then be entered into the standardize menu as further described below. Using this recommended procedure, the online fluoride sensor can be standardized and be in agreement with the grab sampling method chosen without ever having to remove the sensor from process service.

Important Note about 1-point "Standardize" Calibration:

The sensor should be left in service and obtain a stable reading with the process solution. It is not necessary in any way to remove the sensor from service to perform a 1-point grab sample offset "Standardize" calibration.

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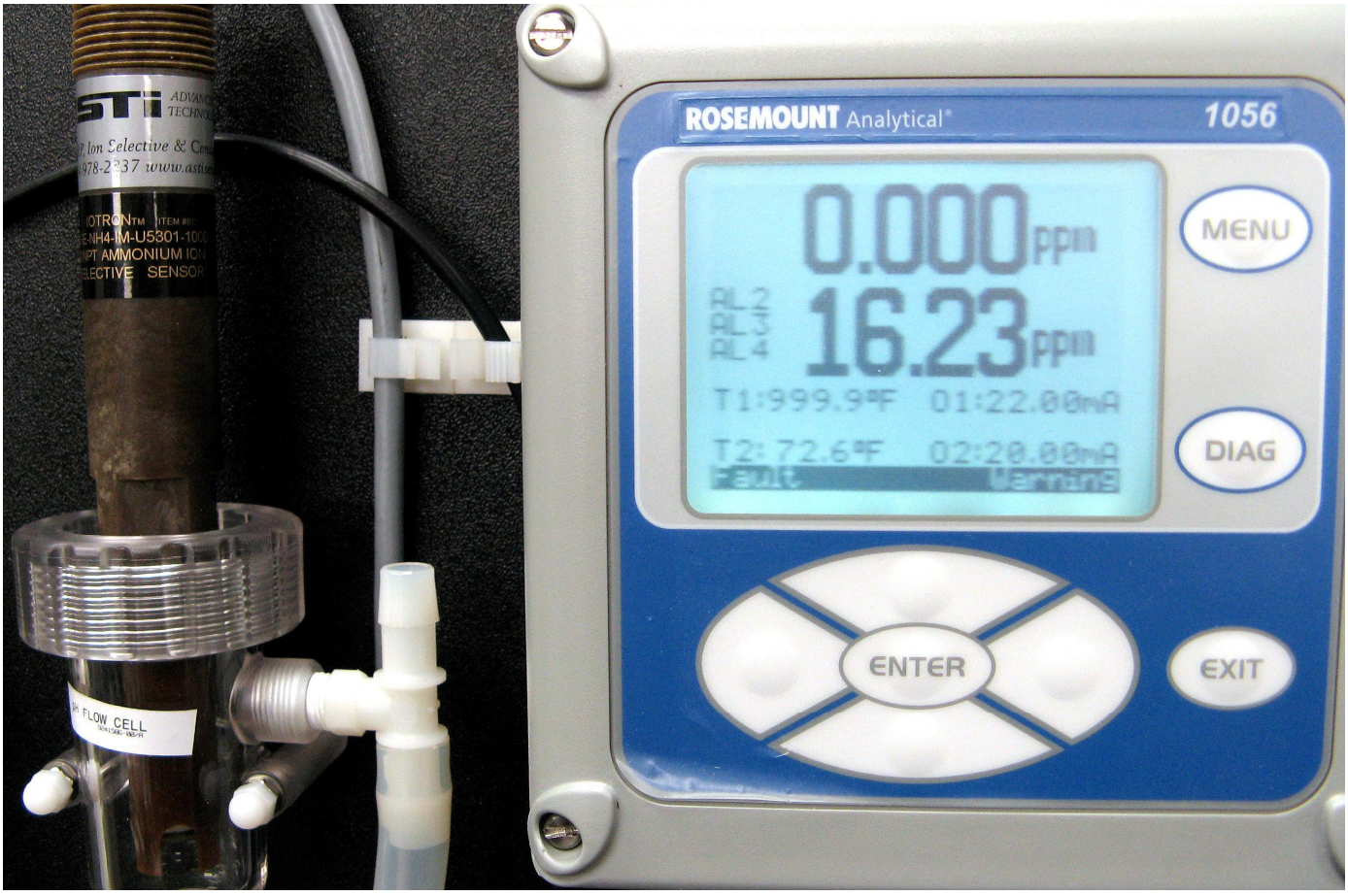
Key Sequence for 1-Point Calibration

1. Press the MENU button
2. Select Calibrate. Press ENTER.
3. Select Sensor 1 or Sensor 2 corresponding to Custom ISE. Press ENTER.
4. Select Standardize Calibration. Press ENTER.
5. Fluoride sensor should be in installed into service with a stable reading.
6. Select edit and enter the ppm value obtained from the grab sample analysis**. This value must be entered even if it already is correct on the display**. After this ppm value has been entered into analyzer, press save.

THE ROSEMOUNT ANALYZER AT THIS POINT SHOULD SNAP TO THE GRAB SAMPLE PPM VALUE ENETERED. IF THIS DOES NOT OCCUR, REPEAT STEPS AS OUTLINED ABOVE UNTIL THE ANALYZER ACCEPTS THE ONE-POINT CALIBRATION.



The low-flow ion selective measurement setup shown above is a representative picture for visualization purposes. Your particular sensor may appear somewhat different to that shown above for a variety of reasons.

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Cleaning and Maintenance of ASTI Fluoride (F-) Probe

Before a major 2-point calibration is performed the sensor may need to be cleaned each time. The frequency of cleaning will depend on the quality of the process water and the build up of process reagents on the probe tip.

Note:

Any noticeable deposits on the tip of the sensor will result in a less accurate calibration and measurement.

CLEANING:

1. Thoroughly rinse the sensor tip with DI water. Gently blot the sensor tip dry.
2. The fluoride sensor tip can be cleaned with isopropyl alochol to remove any 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 or Stanley knife. This reference is solid-state and cannot be damaged with ordinary cleaning techniques. **Do not scratch the membrane.**
4. Once the reference junction has been cleaned the entire sensor tip can be soaked in either the low or high standardization solution. After allowing sufficient time for conditioning before proceed to perform a 2-point calibration.
5. Any calibration 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 standardization solution when not in service in process. The cap should be filled with a calibration standard sealed onto sensor tip with TEFLON tape.

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Miscellaneous

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 organic membrane. This is will cause erroneous readings and 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 cause a noisy signal that may result in erroneous values and calibrations.

Please see the specification and hook-up schematics found in the AB 8100 of AB 6100H fluoride sensor shipping box. This data is included in this ISE addendum for convenience as well in case the hard copy was lost or misplaced.

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.