

Advanced Sensor Technologies, Inc.

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IMPORTANT NOTES FOR FLUORIDE ISE SYSTEM

Calibration and Cleaning of Fluoride Selective Ion Measurement System For Acid Etching Fluoride Analysis

All calibration solutions and Process Grab Sample should be calibrated and tested at identical temperatures to the process temperature to minimize uncertainties associated with the variance of the fluoride activity coefficients with temperature. 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.

Calibration Point 1 (always the lower concentration value) and Calibration Point 2 (always the higher concentration value) determines the response of the 54e Analyzer to a given Fluoride Ion Selective Sensor (AB 6100). The calibration solutions values for Calibration Point 1 is the low ppm value and Calibration Point 2 is high ppm value

The One Point Re-Calibration Standard (usually a process grab sample or one of the two fluoride standard solution) 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.

At all times the calibration solutions should be kept clean and out of direct sunlight and/or other high-energy radiation sources. New sensors should be conditioned in fluoride standard solution for 3 - 5 minutes before beginning calibration procedure as described in this quick calibration guide.

The acceptable pH range of the AB 6100 Fluoride Sensor is 0 – 11, although this kind of sensor has optimal performance in the pH range of 0 - 6. For continuous long-term use, ASTI recommends a pH greater than 1.0 to maximize sensor lifetime. At pH levels below 5.0, 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. To convert the measured fluoride activity (free fluoride) into total fluoride the free fluoride measurement must be compensated for pH. See the attached fluoride dissociation curve for details on how to performance pH compensation on the free fluoride measurement to convert it into free fluoride into total fluoride.

To enter custom ion menu, hold F3 for 5-7 seconds, then enter secret code of 20000 into system.

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

- 1) Enter the nominal ISO Voltage (+ 20 mV) and Slope (- 59.16 mV per decade) as given on the proceeding page.
- 2) Perform a two point calibration to empirically determine slope. See attached procedures for 2-point calibration.
- 3) Perform a one-point calibration (standardize) at the defined (expected) measurement value. See the proceeding section one point calibration for further details on use of this calibration function.
- 4) Place sensor into process and allow to find equilibrium.
- 5) 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 an alternate analysis system, and the online Fluoride system adjusted to read the analyzed value. The sensor should be left in process and this grab sample calibration performed as may be required, unless the sensor seems to be losing sensitivity or giving erratic readings.

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MENU for ROSEMOUNT 54e FLUORIDE ANALYZER

Setting / Value Column	Function / Parameter Column
Main Sub-Menu	Calibrate Main Menu -----
Calibrate Sub-Menu	2-Point Calibration
low ppm	Calibration Point 1 (Factory Defined – DO NOT MODIFY)
high ppm	Calibration Point 2 (Factory Defined – DO NOT MODIFY)
Enabled	Hold Option for Outputs 1 & 2: Read Rosemount 54e-pH/ORP Manual for Details
Calibrate Sub-Menu	Standardize
Variable	Standardize Concentration: Value = Grab Sample, Calibration Solution 1 or 2: See calibration instructions for details on this function; a.k.a. 1-point calibration
Calibrate Sub-Menu	Adjust Temp
User Defined	Manual Adjustment of temperature, not required for most installations
Calibrate Sub-Menu	Temp Compensation
<i>Setting / Value</i>	<i>FUNCTION / PARAMETER</i>
Automatic	Temperature Compensation (Factory Defined – DO NOT MODIFY)
Main Sub-Menu	Diagnostic Variables -----
Defined by 54e	Displays all currently defined variables in 54e Memory
Main Sub-Menu	Program -----
Program Sub-Menu	Alarm Setpoints
Rosemount Default 0.000 ppm & 999.9 ppm	Variable alarm setpoints for 3 @ relays Any user defined value within measurement output range is acceptable
Program Sub-Menu	Output Setpoints
4mA= 0.000 ppm 20mA = 3000 ppm	4-20mA Output 1 – Process Fluoride ISE These are only the factory defaults. These values can be altered.
90 seconds	Time Average of Process Output (Dampening)
4mA= 5°C 20mA = 70°C	4-20mA Output 2 – Temperature These are only the factory defaults. These values can be altered.
20 seconds	Time Average of Temperature Output
Program Sub-Menu	Diagnostics
Disabled	Disabled diagnostics required to make Fluoride ISE measurement run properly (Factory Defined – DO NOT MODIFY)
Program Sub-Menu	Simulated Test
Rosemount Default	Read Rosemount 54e-pH/ORP Manual for Details
Program Sub-Menu	Configure
ASTI Programmed Passcode for Configure	No Passcode has been entered. You may enter a passcode for the configure or program functions to disable unauthorized users from changing any settings
F-	Selective Ion Measurement Parameter (Factory Defined – DO NOT MODIFY)
ppm	Display units for F- Measurement (Defined in Program-Configure Menu)
°C	Display units for Temperature Measurement (Defined in Program-Configure Menu)
-999 to + 999	ISE Input Range in mV's – Rosemount Defined
Custom Ion Curve	Press F3 for 5-7 seconds, enter Passcode of 20000
19.00 grams per mol	IONIC WEIGHT (Defined by Selective Ion Measurement – DO NOT MODIFY)
140 ppm (2.13263)	ISOPOTENTIAL CONCENTRATION (Factory Defined – DO NOT MODIFY)
- 59.16 mV per decade	DEFAULT SLOPE (Will be changed when 2-point calibration is performed)
+ 20 mV	ISOPOTENTIAL VOLTAGE (Will be changed when 2-point calibration is performed)

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Fluoride (F-) Probe Two Point Calibration

This calibration method should not need to be performed frequently. All new fluoride sensors should be calibrated using a 2-point calibration first. Subsequent calibrations can be made using only the standardize option from the Calibrate Main Menu. This calibration determines the sensitivity or slope of each sensor, which is then stored in the 54e transmitter. A “standardize” (a.k.a. 1-point) calibration must be performed after every 2-point calibration. Details for the 1-point calibration are given in the preceding page. **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.
- Low Fluoride Standard Solution (low ppm)
- High Fluoride Standard Solution (high ppm)

Follow the on-screen directions in 2-point calibration submenu in the 54e Analyzer. Calibration Points 1 & 2 have been preprogrammed into your instrument. The low standard solution (low ppm) will always be Calibration Point 1 & the high standard solution (high ppm) will always be Calibration Point 2. Do not change the preprogrammed values for Calibration Point 1 & 2.

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 the attached 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 given below can be used.
- 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 solid state 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 any Function Key
2. Highlight Calibrate and Enter Function
3. Select Hold Mode to On and Continue
4. Select 2-Point Calibration and Enter Function
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 DISPLAY “2-POINT CALIBRATION DONE”. ONLY IF THIS MESSAGE APPEARS HAS THE TWO-POINT CALIBRATION BEEN SUCCESSFULLY PERFORMED, OTHERWISE REPEATED STEPS AS OUTLINED ABOVE UNTIL THE ANALYZER ACCEPTS THE TWO-POINT CALIBRATION.

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Single Point Calibration –

This is the correct method to Adjust for Sensor Drift

Fluoride Sensor One Point Calibration

- Only use the “Standardize” function from the Calibrate Menu. Any other method will destroy the previous 2-point calibration
- Can be performed DAILY if required.

When the fluoride system has been calibrated by the 2-point method it can then be used in the “single point” calibration mode (on a daily basis if required). **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
- Low Fluoride Standard Solution (low ppm) or
- High Fluoride Standard Solution (high ppm) or
- Process Grab Sample Solution

Follow the on-screen directions in the “Standardize” submenu of the Calibration Menu in the 54e Analyzer. No value has been preprogrammed into your instrument for the standardization concentration. Both the low and high standardization solutions can be used to perform the “1-point” calibration performed by the standardize function. In addition, grab samples can be taken from the process and analyzed by an alternate method for ion concentration. The separately determined concentration of the process sample can then be entered into the standardize menu. In this way, the fluoride sensor can be standardized without ever having to remove the sensor from the process line.

Important Notes about 1-point “Standardize” Calibration:

- Fill a 250 mL GLASS beaker with enough standardization solution such that the entire tip of the fluoride sensor will be submersed
- Read the attached 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 given below can be used.
- 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 solid state fluoride ion selective membrane
- Allow a minimum of 3 – 5 minutes for the sensor to stabilize once it has been removed from the tank and placed into either the low or high standard solution.

Key Sequence for 1-Point Calibration

1. Press any Function Key
2. Highlight Calibrate and Enter Function
3. Select Hold Mode to On and Continue
4. Select Standardize and Enter Function
5. Fluoride sensor should be in low ppm standard solution already cleaned and conditioned. Allow reading to stabilize. Press Edit. Enter low ppm into Analyzer and press save. The one point calibration is now complete. The reading on the display should be same as the entered standardize value. Alternatively, the high ppm calibration solution can be used for 1-point calibration if this is expected measurement value. If a grab sample is used, allow the sensor to find a stable reading in process. Enter the analyzed value of the process solution for the fluoride concentration. The sensor should be left in service while this standardize calibration is performed.

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Cleaning and Maintenance of ASTI " F-" Probe

Before a major 2-point calibration 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. Scrape the entire Kynar reference area clean with a sharp blade or Stanley knife. This reference is solid Kynar and cannot be damaged. Do not scratch the membrane.
3. Once this has been achieved the entire tip can be soaked in either the low or high standardization solution. After allowing sufficient time for conditioning; proceed to perform a 2-point or 1-point calibration.
4. The standardization solutions can always be used as conditioning solution for extended storage. Do not allow sensor to be exposed to air for prolonged periods of time. Always store sensor in standardization solution when not in service in process. For long-term storage, a standard solution should be placed into sensor protective cap. The cap should be sealed onto sensor body with Teflon tape.

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Miscellaneous

| The decimal place can be moved in any screen of the 54e 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 solid state membrane. This is will cause erroneous readings and drift.

| The fluoride sensor is comprised of a high-impedance solid state 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 hook-up schematic found in the AB 6100 fluoride sensor shipping box, together with the specification sheet. Be sure to check whether that the jumper in the instrument is set for an internal preamplifier (preamplifier in analyzer setting).