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IMPORTANT NOTES FOR SILVER ISE SYSTEM

Calibration and Cleaning of Silver Selective Ion Measurement System For Waste Water Silver Analysis

All calibration solutions and Process Grab Sample should be calibrated and tested at identical temperatures to the process 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. For greatest overall accuracy of the Silver (Ag+) measurement, however, measurement should be performed as close to 25 degrees Celsius (room temperature process solutions) as possible.

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 Silver Ion Selective Sensor (AB 6170). 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 Silver 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 Silver standard solution for 3 - 5 minutes before beginning calibration procedure as described in this quick calibration guide.

The acceptable pH range of the AB 6170, AB 6170A & AB 8170 Silver Ion Selective Sensor is 1-13.

To enter custom ion menu in the 54epH programmed for Silver ISE Measurement, 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 Silver sensor:

- 1) Enter the nominal ISO Voltage +620 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.
- Place sensor into process and allow it to find electrochemical and thermal 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 Silver 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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INSTRUCTIONS FOR CONFIGURING 1056 ISE ANALYZER for Silver Measurements

For any 1056 ISE Analyzer provided by ASTI the ISE channel MUST be the first channel (Sensor 1). If you have a single channel ISE analyzer (1056-XX-22ISE-38-YY) this allows for only one option, but you should be aware for any future installation that are dual channel ISE/pH analyzers (1056-XX-22ISE-32-YY) and dual channel ISE/conductivity analyzers (1056-XX-22ISE-32-YY) that the ISE sensor should ALWAYS be hooked into channel 1. You should check that your ASTI ISE sensor is properly wired according to the official ASTI hook-up schematic for ASTI sensors with preamplifier or without preamplifiers to the 1056 analyzer:

http://www.astisensor.com/Rosemount 1056 With Preamp Hookup.pdf http://www.astisensor.com/Rosemount 1056 No Preamp Hookup.pdf

The 1056 analyzers will support both ASTI ISE sensors with and without preamplifiers. The ISE channel MUST ALWAYS be set to Custom ISE (If your analyzer shows the measurement for Sensor 1 as anything OTHER THAN Custom ISE <u>IT IS IMPROPERLY CONFIGURED</u>). If your analyzer has a setting of anything other than Custom ISE, it has undoubtedly been 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 below. Normally the user would not need to enter these values 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 (NOT THE SAME AS RESTORE TO ROSEMOUNT FACTORY DEFAULT!!!!)

Custom ISE Variable	Description of Variable	NOTES
107.9 grams per mol	IONIC WEIGHT	Defined by Selective Ion Measurement – DO NOT MODIFY
	Form Wt. in the 1056 analyzer terminology	
0.3340	ISOPOTENTIAL CONCENTRATION	Factory Defined – DO NOT MODIFY
	Iso pCon in the 1056 analyzer terminology	
+59.16 mV per decade	DEFAULT SLOPE	Will be changed when 2-point calibration is performed
	Slope in the 1056 terminology	
+620 mV	ISOPOTENTIAL VOLTAGE	Will be changed when either a 2-point slope or 1-point offset
	Iso Voltage in the 1056 analyzer terminology	standardize calibration is performed

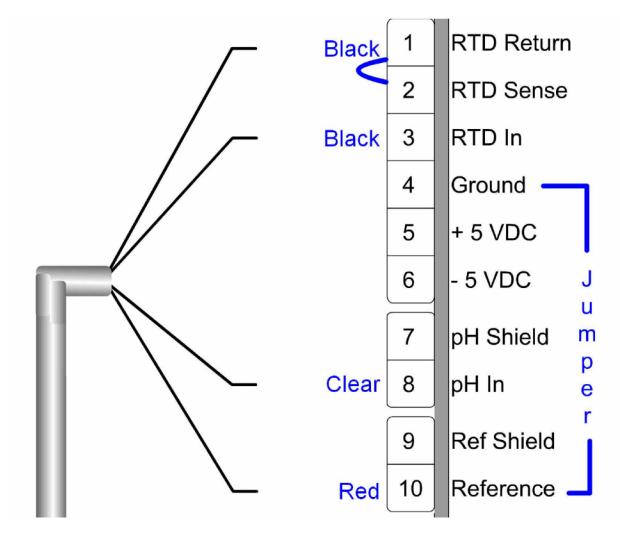
Please note that after restoring the analyzer to the proper Custom ISE configuration (as described above), you will need to repeat your 2-point calibration using calibration solutions that are one decade (10X) apart in value. In addition, you will need to once again place the ISE sensor back into service 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 gram 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 }}



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

Connection from lotron[™] 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).

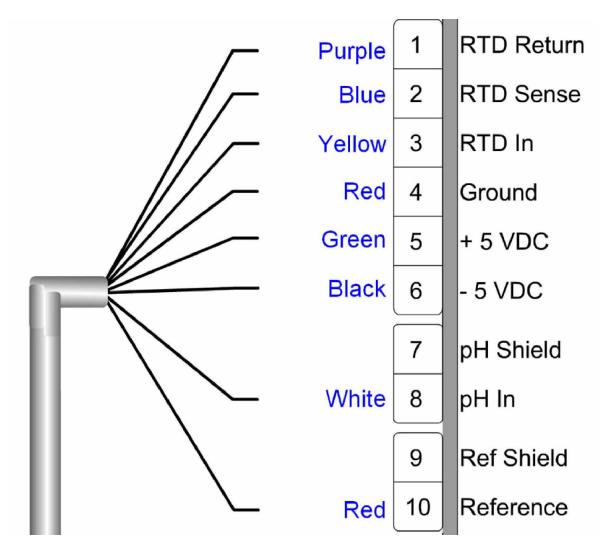
Note 3: Terminals 4 & 10 and terminals 1 & 2 must be tied together to satisfy the analyzer input requirements and disable the reference diagnostic features (pH glass diagnostics should still be available).

Note 4: For Dual Channel Analyzers, please ensure that the proper type of sensor is connected to the proper input board.



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

Connection from lotron[™] 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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MENU for ROSEMOUNT 54e SILVER ANALYZER

Setting / Value Column Function / Parameter Column

Main Sub-Menu		
	Calibrate Main Menu	
Calibrate Sub-Menu	2-Point Calibration	
low ppm	Calibration Point 1	
high ppm	Calibration Point 2	
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	
Setting / Value	FUNCTION / PARAMETER	
Manual 25°C	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	Variable alarm setpoints for 3 @ relays	
0.000 ppm & 999.9 ppm	Any user defined value within measurement output range is acceptable	
Program Sub-Menu	Output Setpoints	
4mA = 0.000 ppm	4-20mA Output 1 – Process Silver ISE	
20mA = 1000 ppm	These are only the factory defaults. These values can be altered.	
90 seconds	Time Average of Process Output (Dampening)	
4mA= 5°C	4-20mA Output 2 – Temperature	
$20\text{mA} = 50 ^{\circ}\text{C}$	These are only the factory defaults. These values can be altered.	
0 seconds	Time Average of Temperature Output	
Program Sub-Menu	Diagnostics	
Disabled	Disabled diagnostics required to make Silver 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	No Passcode has been assigned.	
Passcode for Configure	Various parameters in this function may affect output and display of Analyzer	
Ag+	Selective Ion Measurement Parameter (Factory Defined – DO NOT MODIFY)	
ppm		
°C	Display units for Ag+ Measurement (Defined in Program-Configure Menu)	
-999 to + 999	Display units for Temperature Measurement (Defined in Program-Configure Menu)	
->>> 10 + >>>	ISE Input Range in mV's – Rosemount Defined	
Custom Ion Curve	Press F3 for 5-7 seconds, enter Passcode of 20000	
107.9 grams per mol	IONIC WEIGHT (Defined by Selective Ion Measurement – DO NOT MODIFY)	
0.3340	pISOPOTENTIAL CONCENTRATION (Factory Defined – DO NOT MODIFY)	
+59.16 mV per decade	DEFAULT SLOPE (Will be changed when 2-point calibration is performed)	
+620 mV	ISOPOTENTIAL VOLTAGE (Will be changed when 2-point calibration is performed)	
1 0 20 m v	1 201 012. The Toe Trop (This de Changed when 2 point canoration is performed)	

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Silver (Ag+) Probe Two Point Calibration

This calibration method should not need to be performed frequently. All new Silver 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 proceeding 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 Silver Standard Solution (low ppm)
- High Silver 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 Silver 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 Silver 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. Silver 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. Silver 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 SUCCESFULLY 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

Silver 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 Silver 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 Silver Standard Solution (low ppm) or
- High Silver 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 Silver 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 Silver 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 Silver 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. Silver 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 Silver concentration. The sensor should be left in service while this standardize calibration is performed.

Cleaning and Maintenance of ASTI "Ag+" Probe

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

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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 <u>not</u> 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.
- 5. The Silver ion selective electrode tip may become insensitive to measuring Silver ions due to an exposure to an interfering ion, mechanical damage or exposure to high levels of Silver. In this case, please follow the direction stated in the following pages which are extracted from the technical document Cleaning_Polishing_Solid-State_ISE_Procedure.pdf as posted on our technical support webpages.

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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 Silver ion selective solid state membrane. This is will cause erroneous readings and drift.

The Silver 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 6170, AB 6170A & AB 8170 Silver 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).



pH / ORP / ISE Measurement Product Lines

CLEANING AND POLISHING OF SOLID-STATE IOTRONTM & IOTRODETM ION SELECTIVE ELECTRODE SENSING TIPS

INSPECTION AND CLEANING

Perform the following inspection and polishing procedure when a solid-state ion selective sensor becomes unresponse, sluggish or performing a calibration becomes difficult. The sensor may be contaminated or attacked by some of the components in the sample.

- Remove the sensor from the equipment and visually inspect it. If the sensor's ion selective sensing membrane (the center piece at the front of the sensor) is dull in part or in whole, is recessed into the black body, it may need repolishing.
- If only a deposit of organic nature and is soluble in isopropyl alcohol try to remove by rubbing with a tissue soaked in the alcohol first. If the sensor's sensing membrane regains its shiny state reinsert sensor and recalibrate the equipment per the appropriate ion selective sensor addendum instructions posted at www.astisensor.com/indexrefreshprodoc isemanuals.htm.
- If sensor has been contaminated and contamination cannot be removed with isopropyrl alcohol as described, or the sensing membrane has become dull or recessed into the body, the sensor then needs regrinding and polishing. The sensing membrane is only about 0.100 to 0.150 inches thick, so minimizing the grind thickness allows for a longer service lifetime.

POLISHING

- Use the any "wet or dry 600 grit silicon carbide" polishing paper or cloth for grinding. Place the polishing paper on a smooth surface, and make sure the surface is free from particulates. Wet the polishing surface with high purity deinoized water and hold the sensor perpendicular to the surface of the polishing paper to the middle of the wetted surface.
- Rotate the sensor counterclockwise (clockwise if left handed) while pressing firmly against the surface on an increasing circle to about a 1" to 2" diameter. This motion will partially rough-up the surface of the polishing paper and will rough grind the sensor. Repeat the rotation in reverse by reducing the circle.
- After this procedure the sensor surface should be uniformly dull, showing small scratches. If this does not describe the
 sensor surface, repeat procedure and rotate sensor 180 degrees in your hand. Wash hands and equipment and rinse sensor in
 deionized water.
- Obtain a smooth polishing cloth. Adhere it to a smooth particulate free surface which can be used as a polishing block. Wet the surface of the cloth with deonized water.
- Add about 0.1 gram of 1.0 micron aluminum oxide polishing powder (a fine white powder). Use the same technique and motions for polishing as described in the grinding section, starting in the middle of the aluminum oxide pool.
- Check the surface after about six to ten circles, if not shiny and scratch free to the naked eye, repeat the procedure. Rotate the sensor 180 degrees in your hand after each checking as this will provide more uniform surface.
- Wash hands and equipment, rinse sensor in deonized water. Condition the sensor for about 30 minutes in the calibration solution of the lower concentration and recalibrate the equipment as described in the ion selective addendum.
- If the polishing cloth is cleaned and stored clean it can be repeatedly used, if broken down, replace with new one.