Advanced Sensor Technologies, Inc.
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IMPORTANT NOTES FOR CALCIUM ISE SYSTEM FOR HIGH RANGE APPLICATIONS

Calibration and Cleaning of Calcium Selective Ion Measurement System For Calcium Analysis in High Sodium and Strong Ionic Strength Solutions

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. The valid (permissible) temperature range for all calcium ion selective sensors is five to forty (5-40) degrees Celsius (41 to 104 degrees Fahrenheit).

The primary calibration method is a 1-point grab sample offset to create agreement between the inline sensor reading and the reference analysis method (typically a portable photometer). This 1-point grab sample offset calibration is done with the sensor left in service after it is sufficiently equilibrated with the process stream. For most new systems no 2-point slope calibration is required at all. If you feel that a 2-point slope calibration is required, suitable calibration standard need to be prepared. Please see last page of this addendum for the procedures to fabricate such suitable calibration standards. If you are not sure of the most suitable calibration standard for your particular application, please contact the ASTI factory.

Calibration Point 1 is always the lower concentration calcium value and Calibration Point 2 is always the higher calcium concentration value. Using these two calibration standards in the 2-point calibration mode determines the characteristic response (slope) of any given calcium Ion Selective Sensor (AB 6440, AB 6440A and AB 8440). 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 values. New sensors should be conditioned in calcium standard solution or process media for a sufficient period of time before beginning any of the calibration procedures as described in this guide.

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The acceptable pH range of the AB 6440, AB 6440A and AB 8440 calcium ion selective sensors is 2 to 12 at 25 degrees Celsius. For optimal performance ASTI recommends a pH between 4 and 10 at any temperature from 5 to 40 degrees Celsius.

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

1) Enter the nominal ISO Voltage +98.59 mV and Slope (+25.03 mV per decade)
2) 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.
3) 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 (such as a portable colorimeter) and the online calcium 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.

OPTIONAL FOR OLDER SENSORS:

4) It is possible to perform a two-point calibration to empirically determine slope. In most cases the empirical determination of the slope is not at all necessary and the ASTI factory recommended characteristic slope is best as setup at time of dispatch. You should contact the ASTI factory before performing any 2-point slope calibration to ensure best results. Note that a 1-point offset calibration MUST still be performed after any 2-point slope calibration is done.

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Configuring 1056 Analyzers for Calcium Measurement

Please 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_1057_56_No_Preamp_Hookup.pdf

http://www.astisensor.com/Rosemount_1056_1057_56_With_Preamp_Hookup.pdf

The 1056 ISE 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 IT IS IMPROPERLY CONFIGURED). 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 back into Custom ISE mode:

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

Enter the following four values into the Custom ISE setup menu:

Custom ISE Variable	**Description of Variable**	**NOTES**
40.08 grams per mol	IONIC WEIGHT **Form Wt.** in the 1056 analyzer terminology	Defined by Selective Ion Measurement – DO NOT MODIFY
3.50937	ISOPOTENTIAL CONCENTRATION **Iso pCon** in the 1056 analyzer terminology	Factory Defined – DO NOT MODIFY
+25.03 mV per decade	DEFAULT SLOPE **Slope** in the 1056 terminology	Will be changed when 2-point calibration is performed
+98.59 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

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.

There are a few additional settings that will also need to changed:

Main Menu -> Program -> Measurement -> Sensor 1 -> Reference "Z"

Change from the settings for the reference "Z" to "High"

Main Menu -> Calibrate -> Sensor 1 -> Custom ISE -> Setup

Change Stabilize Time from 10 seconds (default) to 30 seconds.

Change Stabilize Delta from 1mV (default) to 3mV.

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!!!!).

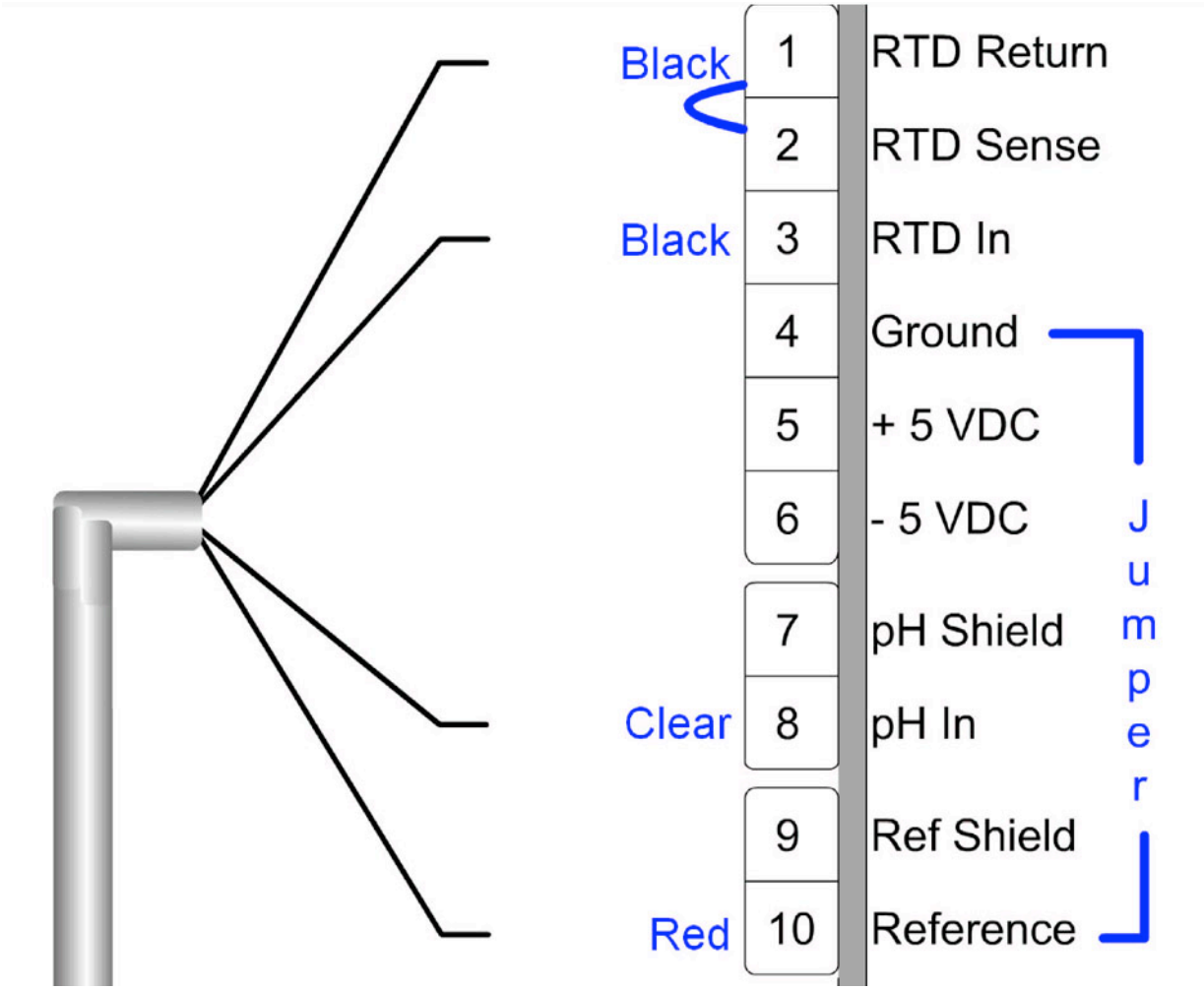
Please note that after restoring the analyzer to the proper Custom ISE configuration (as described above), you will then also need to repeat your 1-point grab sample calibration (standardize in the 1056 terminology). This means taking a grab sample from the process and determining the ISE concentration and then using the 1-point standardize calibration 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 }}

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).

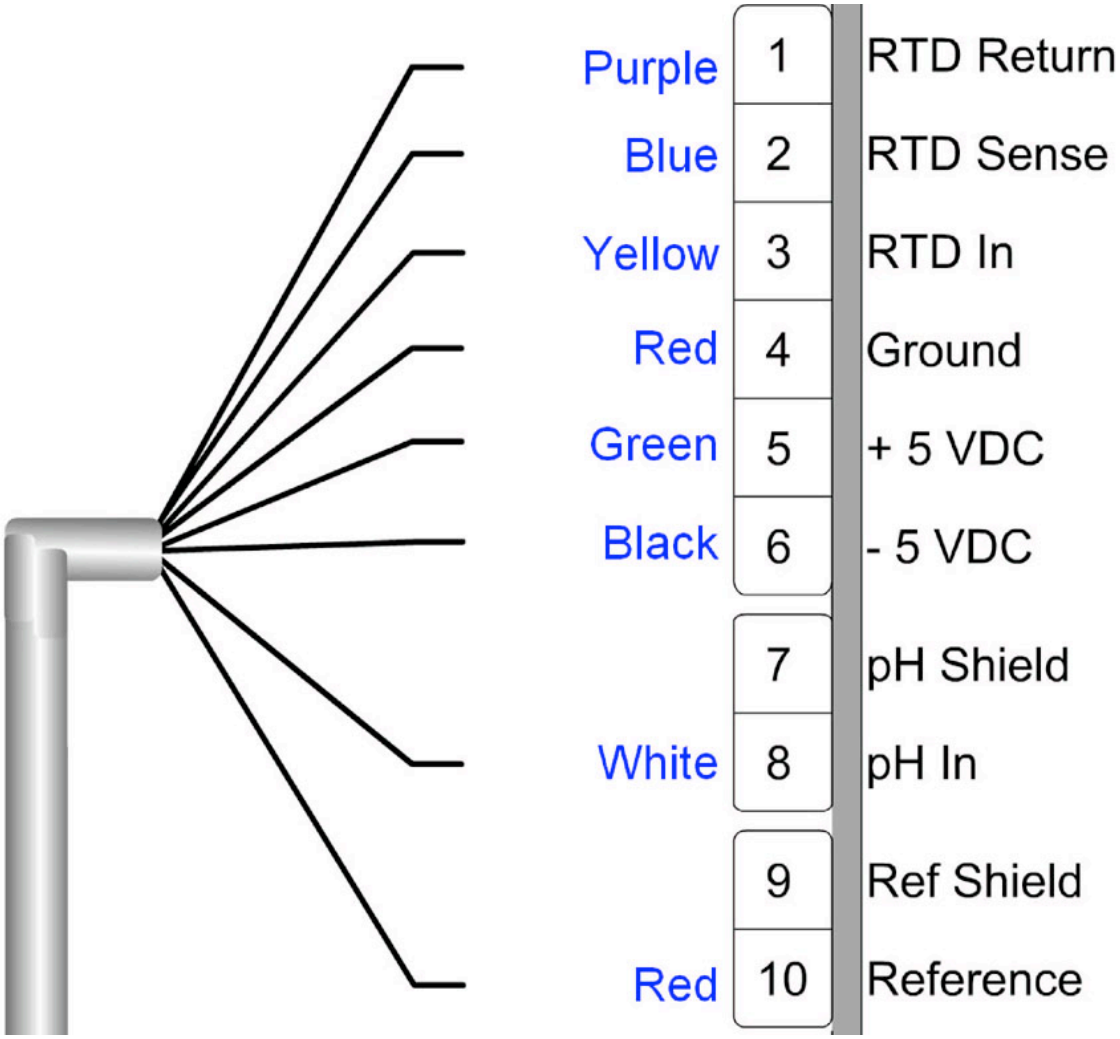
Note 2: For ORP and Ion Selective Sensors, please put the active signal (clear) to terminal 8 (indicated as pH In).

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.

Connection Diagram of Iotron™ pH / ORP / ISE Sensors **With** 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: 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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Calcium (Ca^{++}) Probe Two Point Calibration

This calibration method should not need to be performed frequently and new calcium ISE sensor do not generally need a 2-point slope calibration to be performed at all (ready for installation directly from the factory). This two-point calibration determines the sensitivity or slope of each sensor, which is then stored in the analyzer. Subsequent 1-point offset calibrations then needs to be made using 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 pages. **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 or 500 mL GLASS OR PLASTIC BEAKERS
(Preferably heavy enough so that the ISE sensor does not tip over the beaker)
Low Calcium Standard Solution
High Calcium Standard Solution

Follow the on-screen directions in 2-point calibration submenu in the 1056 Analyzer. The low standard solution will always be Calibration Point 1 & the high standard solution will always be Calibration Point 2. The exact values for these low and high calcium standard solutions will depend upon your exact needs as will the compositions of the standards depending upon your intended application use. Please review the last page

Important Notes about Calibration:

- Fill a 250 mL GLASS beaker with enough standardization solution such that the entire tip of the Calcium 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 calcium 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.
- Allow a minimum of 3 – 5 minutes for the sensor to stabilize between the low and high Calibration Solutions.

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 the Custom ISE. Press ENTER.
5. Select Custom ISE, Press ENTER.
6. Select 2-point Calibration. Press ENTER.
7. Calcium sensor should be in low ppm or % standard solution already cleaned and conditioned. Select Standard 1 and press ENTER. Instrument will display that Calibration Point 1 is stabilizing. The instrument will take about 30 seconds to stabilize and determine mV value for the first calibration point.
8. After Cal Point 1 has stabilized select edit and input low ppm or % into instrument**. This value must be entered even if it already is correct on the display**. After low ppm or % has been entered into analyzer, press save to continue to second calibration point.
9. Calcium sensor should be in high ppm or % standard solution already cleaned and conditioned. Select Standard 2 and press ENTER. Instrument will display that Calibration Point 2 is stabilizing. The instrument will take about 30 seconds to stabilize and determine mV value for the second calibration point.
10. After Cal Point 2 has stabilized select edit and input high ppm or % into instrument. **This value must be entered even if it already is correct on the display.** After high ppm or % has been entered into analyzer, press save.

THE ROSEMOUNT ANALYZER AT THIS POINT SHOULD SNAP TO THE NOMINAL PPM OR % VALUE OF THE HIGH CALCIUM PPM OR % 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.

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

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

Calcium Sensor One Point Calibration ("Standardize")

Only use "Standardize" function in Calibrate Menu.

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

When the calcium sensor has been calibrated by the 2-point method previously described or the default factory slope value is to be used, 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 1056 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 calcium concentration (typically a portable colorimeter). This grab sample determined concentration of the process sample will then be entered into the standardize menu as further described below. Using this recommended procedure, the online calcium 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 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 the Custom ISE. Press ENTER.
4. Select Custom ISE, Press ENTER.
5. Select Standardize. Press ENTER.
6. Calcium sensor should be in installed into service with a stable reading.
7. Select edit and enter the ppm or % value obtained from the grab sample analysis**. This value must be entered even if it already is correct on the display**. After this ppm or % value has been entered into analyzer, press save.

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

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Cleaning and Maintenance of ASTI Calcium (Ca++) 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. 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.**
3. Once the reference junction has been cleaned the entire sensor tip can be soaked in either the low or high standardization solution or else installed back into service for a 1-point grab sample offset calibration. Be sure to allow sufficient time for conditioning before proceed to perform a 1-point of 2-point calibration.
4. 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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Web Site: http://www.astisensor.com Technical Support: http://www.astisensor.com/cgi-bin/ttx.cgi

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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 calcium ion selective organic membrane. This is will cause erroneous readings and drift.

- The calcium 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 6440, AB 6440A and AB 8440 calcium ion selective 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 High Calcium Standard Solutions

Materials

Calcium Chloride Anhydrous - CAS # 10043-52-4 (Analytical/Reagent Grade or better, new sealed dry bottle preferred)
Sodium Chloride - CAS # 7440-23-5 (Analytical/Reagent Grade or better, brand new sealed dry bottle preferred)
1 Liter Volumetric Flask (2 each)
5 mL volumetric pippete
50 mL volumetric pippete
5 liter plastic bottles with air-tight sealing cap (5 each)
DI Water (15 MegaOhms or higher resistivity grade)

- 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 10,000 ppm Calcium stock solution:

1. Measure out 27.692 grams of Calcium chloride salt.
2. Place this Calcium Calcium into 1 liter volumetric flask.
3. Dilute with DI water to the 1 liter mark. Mix solution well until it is completely homogeneous.
4. Transfer this 10,000 ppm Calcium stock solution to a 1 liter plastic bottle and label appropriately.

Preparation of 10,000 ppm Sodium stock solution ***(REPEAT THIS PROCEDURE TWICE)***

5. Measure out 25.421 grams of sodium chloride salt.
6. Place this sodium sodium into 1 liter volumetric flask.
7. Dilute with DI water to the 1 liter mark. Mix solution well until it is completely homogeneous.
8. Transfer this 10,000 ppm sodium stock solution to a 1 liter plastic bottle and label appropriately.

Calcium Calibration Solution Preparation Procedures:

Preparation of 50 ppm Calcium Standard Ion Solution

1. Draw 5.0 mL of 10,000 ppm Calcium stock solution and transfer to a 1 liter volumetric flask.
2. Dilute with 10,000ppm sodium chloride stock solution to the 1 liter mark. Mix solution well until it is completely homogeneous.
3. Transfer this 50 ppm Calcium calibration solution to a 1 liter plastic bottle, seal and label appropriately.

Preparation of 500 ppm Calcium Standard Ion Solution

4. Draw 50.0 mL of 10,000 ppm Calcium stock solution and transfer to a 1 liter volumetric flask.
5. Dilute with 10,000ppm sodium chloride stock solution to the 1 liter mark. Mix solution well until it is completely homogeneous.
6. Transfer this 500 ppm Calcium calibration solution to a 1 liter plastic bottle, seal and label appropriately.