

# *Advanced Sensor Technologies, Inc.*

TEL: 714-978-2837 TOLL FREE: 1-888-WOW-ASTI (969-2784) FAX: 714-978-6339

## **IMPORTANT NOTES FOR AMMONIUM ISE SYSTEM**

### **Calibration and Cleaning of Ammonium Selective Ion Measurement System**

#### **For ULTRA-HIGH LEVEL MEASUREMENTS ~ 1,000 TO 50,000 PPM (0.1 TO 5.0%)**

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](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 ammonium ( $\text{NH}_4^+$ ) 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 ammonium ion selective sensors is five to forty (5-40) degrees Celsius (41 to 104 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 ammonium Ion Selective Sensor (AB 6410, AB 6410A and AB 8410). 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 ammonium standard solution for 3 - 5 minutes before beginning the calibration procedure as described in this guide.



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## **Configuring 1056 Analyzers for Ammonium Measurement**

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) 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_With_Preamp_Hookup.pdf)

[http://www.astisensor.com/Rosemount\\_1056\\_No\\_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 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:

***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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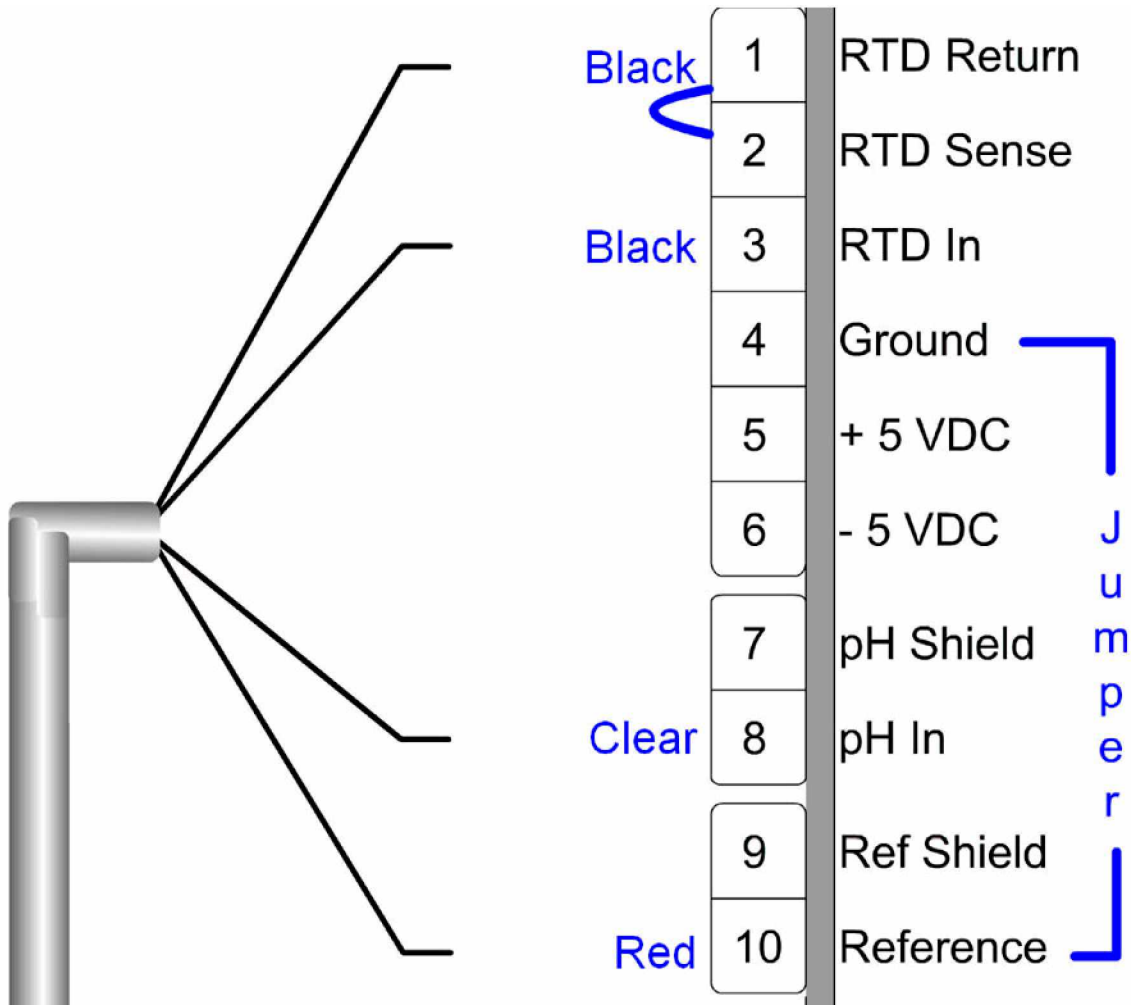
Web Site: <http://www.astisensor.com> Technical Support: <http://www.astisensor.com/cgi-bin/ttx.cgi>





## 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).

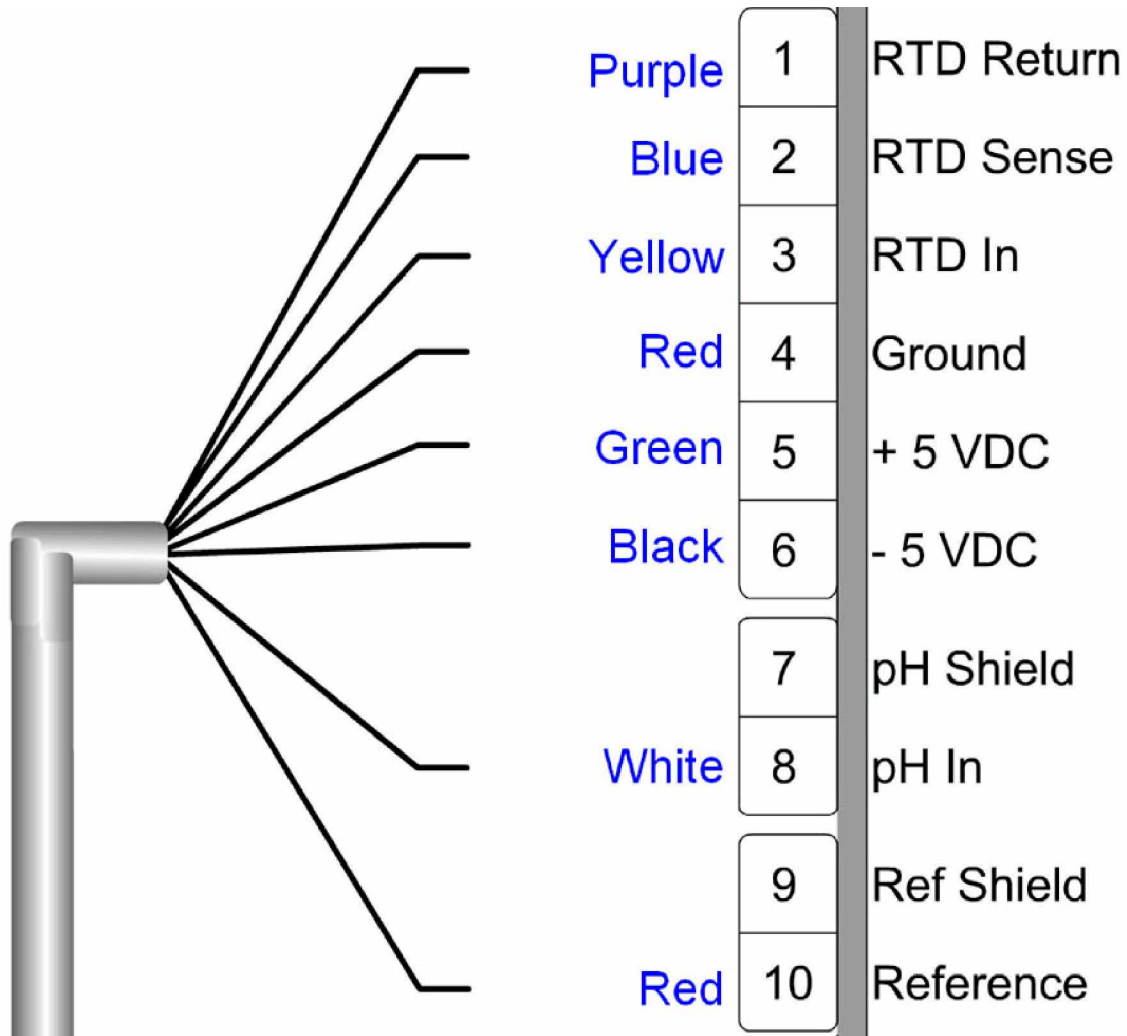
Note 2: For ORP and Ion Selective Sensors, please put the active signal (clear) to terminal 8 (indicated as pH In).

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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## **Ammonium (NH<sub>4</sub><sup>+</sup>) Probe Two Point Calibration**

This calibration method should not need to be performed frequently. All new ammonium 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 Ammonium Standard Solution (low ppm)

High Ammonium 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 ammonium standard solutions will depend upon your exact needs. The USA Blue book are commonly used to calibrate our ammonium ion selective sensors and a link is provided below to the relevant page of the catalog (it is also included in this addendum for convenience).

<http://www.astisensor.com/Calibration/Ammonia Standards USA Blue Book.pdf>

There are some incorrect naming conventions that have persisted over the years that have unfortunately led to confusion with a number of customers. For example the USA Blue Book ammonia standards are indicated as NH<sub>3</sub>-N such that the ppm values given are valid only for nitrogen contribution. To convert to ammonium ppm units (required for performing a two-point slope calibration with the 1056 analyzer), simply multiply the NH<sub>3</sub>-N ppm concentration given by 1.287844.

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Web Site: <http://www.astisensor.com> Technical Support: <http://www.astisensor.com/cgi-bin/ttx.cgi>

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For convenience, find below the typical USA Blue book standards converted from ppm NH<sub>3</sub>-N to ppm ammonium. These ppm ammonium values should be the units entered into the 1056 ISE analyzer. These standards are quite popular as they are low cost, readily available and suitable for the purposes of obtaining a good two-point calibration slope.

1 ppm NH <sub>3</sub> -N	1.29 ppm ammonium (NH <sub>4</sub> <sup>+</sup> )
10 ppm NH <sub>3</sub> -N	12.88 ppm ammonium (NH <sub>4</sub> <sup>+</sup> )
100 ppm NH <sub>3</sub> -N	128.78 ppm ammonium (NH <sub>4</sub> <sup>+</sup> )
1000 ppm NH <sub>3</sub> -N	1287.84 ppm ammonium (NH <sub>4</sub> <sup>+</sup> )

Alternatively, instructions on how to prepare your own ammonium calibration standards using the typical in-house ASTI fabrication procedure can be obtained by using the URL link below. These procedures are also included in this addendum for convenience. Either the USA Blue Book or ASTI procedure prepared standards will allow you to successfully perform a two-point slope calibration.

[http://www.astisensor.com/Calibration/Ammonium\\_Calibration\\_Solutions\\_Preparation\\_Procedure\\_06-10-05.pdf](http://www.astisensor.com/Calibration/Ammonium_Calibration_Solutions_Preparation_Procedure_06-10-05.pdf)

## **Important Notes about Calibration:**

- Fill a 250 mL GLASS beaker with enough standardization solution such that the entire tip of the Ammonium 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 ammonium 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).

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## 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. Ammonium sensor should be in low ppm 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 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.
9. Ammonium sensor should be in high ppm 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 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 AMMONIUM 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.**

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

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

### **Ammonium Sensor One Point Calibration (“Standardize”)**

Only use “Standardize” function in Calibrate Menu.

Can be performed as frequently as may be required.

When the ammonium 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 ammonia 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 ammonium 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 the Custom ISE. Press ENTER.
4. Select Custom ISE, Press ENTER.
5. Select Standardize. Press ENTER.
6. Ammonium sensor should be in installed into service with a stable reading.
7. 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.**

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## **Cleaning and Maintenance of ASTI Ammonium (NH<sub>4</sub><sup>+</sup>) 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. After allowing sufficient time for conditioning before proceed to perform a 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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## **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 ammonium ion selective organic membrane. This is will cause erroneous readings and drift.
- The ammonium 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 6410, AB 6410A and AB 8410 ammonium 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.

**Alkalinity Titrant, Sulfuric Acid, 0.02 N**

APHA for Alkalinity (2320-B)

SIZE	CONTAINER	STOCK #	EACH
500 mL	Poly	MB-29848	\$ 6.78
1 L	Poly	MB-29849	10.04
4 L	Poly	MB-29850	21.70

**Ammonia ISA Buffer**

For use with Ammonia ISE

SIZE	CONTAINER	STOCK #	EACH
500 mL	Poly	MB-29448	\$ 18.86
1 L	Poly	MB-29449	31.43

**Ammonia Standard Solution as N, 1.0 mg/L (ppm)**

SIZE	CONTAINER	STOCK #	EACH
500 mL	Poly	MB-29450	\$ 13.04
1 L	Poly	MB-29451	18.91

**Ammonia Standard Solution as N, 10 mg/L (ppm)**

SIZE	CONTAINER	STOCK #	EACH
500 mL	Poly	MB-29452	\$ 13.04
1 L	Poly	MB-29453	18.91

**Ammonia Standard Solution as N, 100 mg/L (ppm)**

SIZE	CONTAINER	STOCK #	EACH
500 mL	Poly	MB-29454	\$ 14.67
1 L	Poly	MB-29455	19.56

**Ammonia Standard Solution as N, 1000 mg/L (ppm)**

SIZE	CONTAINER	STOCK #	EACH
500 mL	Poly	MB-29456	\$ 17.39
1 L	Poly	MB-29457	23.92

**Ammonium Chloride Solution, 1.15 g/L**

APHA for BOD (5210-B)

SIZE	CONTAINER	STOCK #	EACH
500 mL	Poly	MB-29461	\$ 10.61

**Ammonium Chloride, ACS Grade**

CAS No. 12125-02-9

SIZE	CONTAINER	STOCK #	EACH
100 g	Poly	MB-29462	\$ 9.32
500 g	Poly	MB-29463	23.91

**Ammonium Hydroxide, ACS Grade**

(28-30% Aqua Ammonia)

CAS No. 1336-21-6

SIZE	CONTAINER	STOCK #	EACH
500 mL	Glass	MB-29464	\$ 13.26
1 L	Glass	MB-29465	19.55
2.5 L	Glass	MB-29466	28.76

**Ammonium Hydroxide, 3%**

(Use to clean silver anode on YSI DO probe)

SIZE	CONTAINER	STOCK #	EACH
500 mL	Poly	MB-29468	\$ 9.32
1 L	Poly	MB-29469	15.54

**Ammonium Molybdate Tetrahydrate, ACS Grade**

CAS No. 12054-85-2

 $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}$  FW= 1235.86

SIZE	CONTAINER	STOCK #	EACH
100 g	Poly	MB-29470	\$ 30.38
500 g	Poly	MB-29471	76.98

**Ammonium Molybdate, 4% (w/v)**

APHA for Phosphorus (4500-E, F)

SIZE	CONTAINER	STOCK #	EACH
500 mL	Poly	MB-29473	\$ 21.73

**Ammonium Persulfate, ACS Grade**

CAS No. 7727-54-0

 $(\text{NH}_4)_2\text{S}_2\text{O}_8$  FW= 228.20

SIZE	CONTAINER	STOCK #	EACH
100 g	Poly	MB-29476	\$ 12.43
500 g	Poly	MB-29477	28.68

**Ascorbic Acid, ACS Grade**

CAS No. 50-81-7

 $\text{C}_6\text{H}_8\text{O}_6$  FW= 176.13

SIZE	CONTAINER	STOCK #	EACH
100 g	Poly	MB-29479	\$ 16.97
500 g	Poly	MB-29480	48.95

**Concentration Conversions**

Multiply	→	to get
to get	←	Divide
mg/L	1	ppm (when specific gravity=1.00)
g/L	1000	mg/L
mg/L	1000	µg/L

operator notes

**Example:** If your concentration is 20 g/L, multiplying by 1000 shows your concentration in mg/L: 20,000 mg/L. If your concentration is 2000 µg/L, dividing by 1000 shows your concentration in mg/L: 2 mg/L.

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## Procedures for Preparation of Ammonium Standard Solutions

### Materials

Ammonium Chloride - CAS # 12125-02-9 (Analytical/Reagent Grade or better, brand new sealed dry bottle preferred)  
Magnesium Chloride Hexahydrate - CAS # 7791-18-6 (Commercial Grade OK)  
1 Liter Volumetric Flask (one each)  
5 Liter Volumetric Flask (one each)  
1 liter plastic bottles (five each)  
1 mL volumetric pipette  
10 mL volumetric pipette  
1 liter opaque plastic bottles with air-tight sealing cap (five 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 0.10 Molar Magnesium Chloride stock solution (DO THIS FIRST!):*

1. Measure out 20.33 grams of magnesium chloride hexahydrate.
2. Place this magnesium chloride into a 5 liter volumetric flask.
3. Dilute with DI water to 5 liter mark. Mix solution well until all magnesium chloride is dissolved.
4. Seal 5 liter volumetric flask with glass stopper.

#### *Preparation of 10,000 ppm Ammonium stock solution:*

1. Measure out 29.654 grams of ammonium chloride salt.
2. Place this ammonium chloride 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 ammonium stock solution to a 1 liter plastic bottle and label appropriately.

### Ammonium Calibration Solution Preparation Procedures:

#### *Preparation of 50 ppm Ammonium Standard Ion Solution*

1. Draw 5.0 mL of 10,000 ppm ammonium stock solution and transfer to a 1 liter volumetric flask.
2. Dilute with 0.10 Molar magnesium chloride stock solution to the 1 liter mark. Mix solution well until it is completely homogeneous.
3. Transfer this 50 ppm ammonium calibration solution to a 1 liter plastic bottle and label appropriately.

#### *Preparation of 500 ppm Ammonium Standard Ion Solution*

4. Draw 50.0 mL of 10,000 ppm ammonium stock solution and transfer to a 1 liter volumetric flask.
5. Dilute with 0.10 Molar magnesium chloride stock solution to the 1 liter mark. Mix solution well until it is completely homogeneous.
6. Transfer this 500 ppm ammonium calibration solution to a 1 liter plastic bottle and label appropriately.