

AMMONIA IN SOLUTIONS CONTAINING FATS, FATTY ACIDS, OILS, AND SURFACTANTS

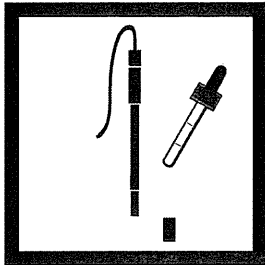
The Air Gap Accessory optimizes performance and extends the usable lifetime of the membrane module. Samples containing substances known to cause membrane failure (e.g., surfactants, oils, fats) can be measured repeatedly with this accessory. Slopes, absolute mV readings, and response times may change significantly when the Air Gap Accessory is used. Therefore, use the Air Gap Accessory for all calibrations and measurements.

Principle of Operation

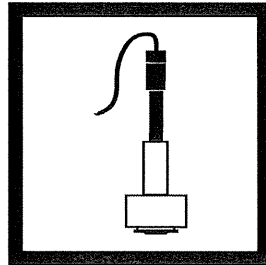
The Ammonia Electrode uses a fine, porous membrane to separate the sample solution from a thin layer of electrolyte that coats the pH sensing bulb inside the electrode. Ammonia gas can diffuse through, but other ions may not. When the Air Gap Accessory is used, substances that can foul the membrane are separated from the membrane by a small air space. Ammonia gas diffuses through the air space and then through the membrane. The air space has slightly different diffusion characteristics than the membrane, which causes an increased response time.

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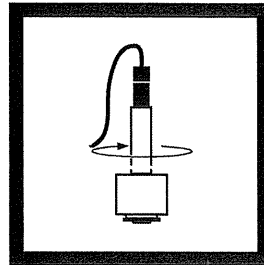
Assembling the Air Gap Accessory



1. Fill a membrane module and attach it to the Ammonia Electrode as described in *Section 1.2* on page 10. Condition the electrode in Ammonia Electrode Storage Solution for at least 15 minutes.



2. Slide the Ammonia Electrode into the air gap electrode holder, starting from the narrow end of the electrode holder.



3. With a clockwise twist, push the electrode through the internal O-ring until the electrode cap touches the top of the electrode holder. The electrode normally stops about 0.25 inches from the top of the electrode holder because of the O-ring. Push the electrode through the O-ring.

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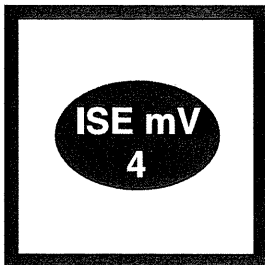
Calibration



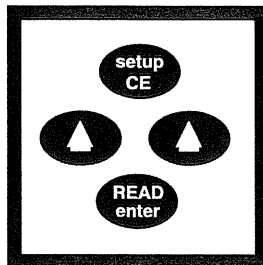
1. Connect the Ammonia ISE to the *sensION™4* pH/ISE meter. Verify that BNC is selected in Setup 1 of the Setup menu.

Note: Only the *sensION4* meter is recommended for this method.

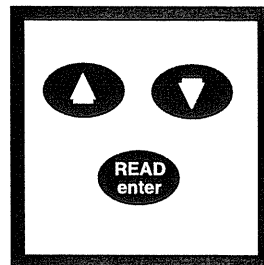
Note: The *sensION4* has two BNC and two five-pin connectors. Choose one of the BNC connectors for the ammonia electrode.



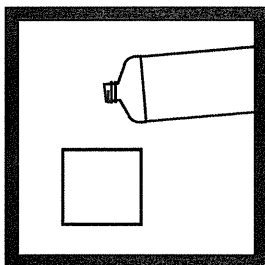
2. Turn the meter on. Press **ISE/mV** until the display shows **mg/L** (or user selected concentration units).



3. Press **SETUP**. Press the Up arrow until the display shows **Stabilizing...** and a mV value.

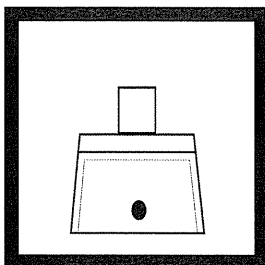


4. Press **ENTER** and use the numerical keys to edit the display until it shows **0.1 mV/min**. Press **ENTER**, then press **EXIT**.



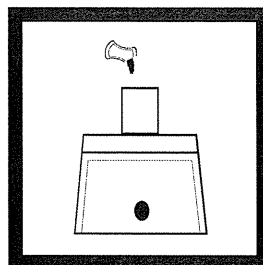
5. Fill the Air Gap Accessory sample cup to the line with 1.0-mg/L $\text{NH}_3\text{-N}$ standard.

Fill another sample cup to the line with 10-mg/L $\text{NH}_3\text{-N}$ standard.

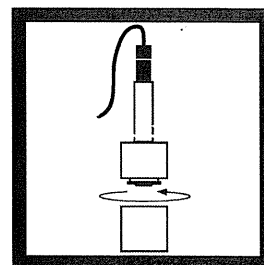


6. Add a stir bar to each cup. Place the cup the 1.0-mg/L standard on a stirrer. Stir at a moderate rate. When the speed is properly adjusted, turn the motor off.

Note: Stir so there is **slight** vortexing of the sample in the cup.

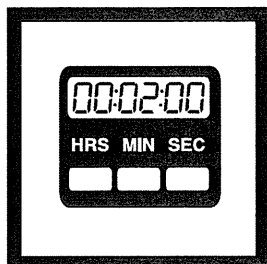


7. Add the contents of one Ammonia Ionic Strength Adjustor powder pillow to the cup on the stirrer. Do not stir.

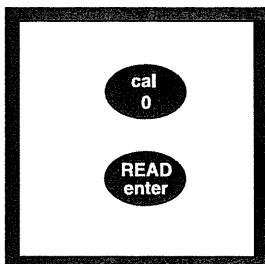


8. Holding the cup firmly with one hand, carefully insert the electrode holder into the sample cup with a slight twist to set the O-ring. Bubbling at the air vent may occur.

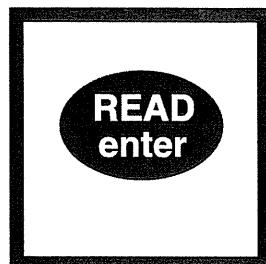
AMMONIA IN SOLUTIONS CONTAINING FATS, FATTY ACIDS, OILS, AND SURFACTANTS, continued



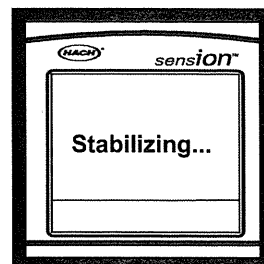
9. Turn the stirrer on and stir for 2 minutes.



10. After the two minute period, press **CAL**. The display will show **CAL, ?, mg/L**, and the active keys. The units will be flashing. Use the arrow keys to select the desired units, then press **ENTER**.

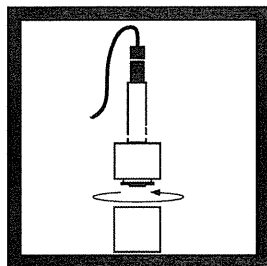


11. The display will show **Standard 1, ?**, and the value of standard 1 from the previous calibration. Press **ENTER** to accept the numerical value or use the number keys to change the value to match the 1.0-mg/L concentration of the standard, then press **ENTER**.

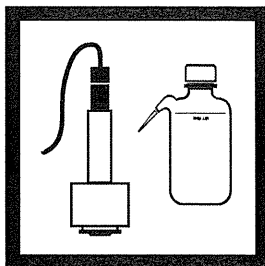


12. The display will show **Stabilizing...** until the reading is stable. The display will then show **---** or the value of standard 2 from the previous calibration.

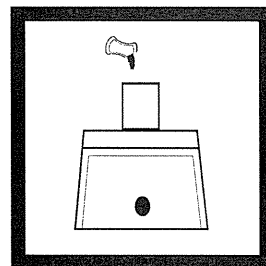
Note: It may take up to five minutes for the electrode to stabilize.



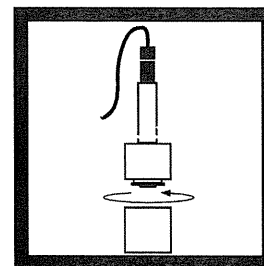
13. Remove the electrode holder assembly by holding the sample with one hand on a horizontal surface and pulling out with a slight twist. Shake any excess sample off the holder while keeping the holder in a vertical position.



14. Rinse the electrode holder with deionized water, including the inside sample reservoir via the reservoir vent. Gently blot the entire holder. Turn the stirrer off.

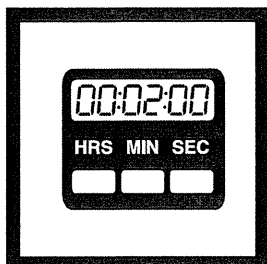


15. Place the 10-mg/L standard on the stirrer. Add the contents of one Ammonia Ionic Strength Adjustor powder pillow to the cup on the stirrer. Do not stir.

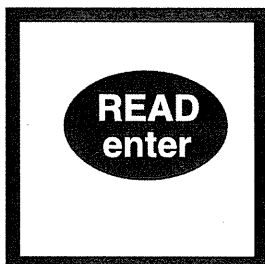


16. Holding the cup firmly with one hand, carefully insert the electrode holder into the sample cup with a slight twist to set the O-ring. Bubbling at the air vent may occur.

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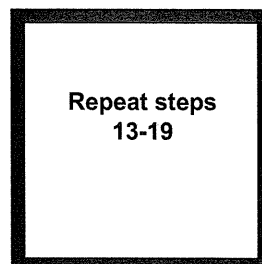
17. Turn the stirrer on and stir for 2 minutes.



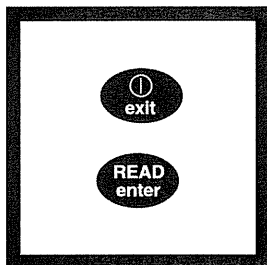
18. Press **ENTER** to accept the displayed value for standard 2 or use the numerical keys to change the value to 10.0 mg/L, then press **ENTER**.



19. The display will show **Stabilizing...** until the reading is stable. The display will then show $____$ or the value of standard 3 from the previous calibration.

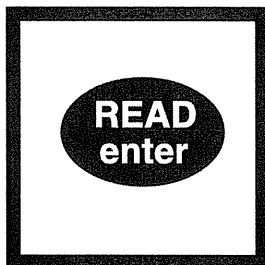


20. Repeat *steps 13-19* for each additional standard.



21. After the last standard is measured, press **EXIT**.

Note: After the seventh standard, the meter automatically displays the **STORE** prompt.

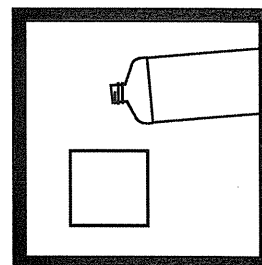


22. The display will show **Store?**. Press **ENTER** to store the calibration or **EXIT** to leave the calibration mode without storing the calibration values.



23. Press **REVIEW**. Use the Up arrow key to scroll to the last slope value. It should be -57 ± 3 mV/decade. Press **EXIT** to return to measurement mode.

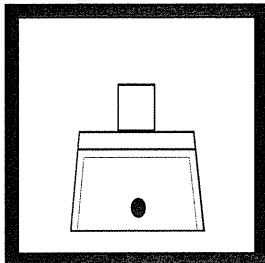
Note: If the slope is not approximate -57 ± 3 mV/decade, recalibration may be necessary. If the slope is still incorrect after recalibration, replace the ammonia module.



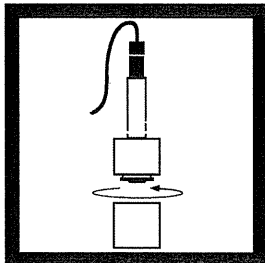
24. Fill a sample cup to the line with sample. Add a stir bar.

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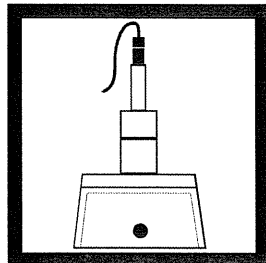
Sample Measurement



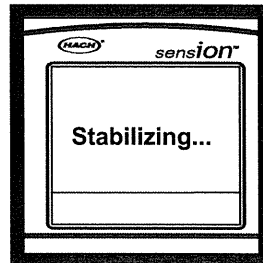
25. Place the cup on the stirrer and stir at the same speed as the standards. When the speed is adjusted correctly, turn the stirrer off.



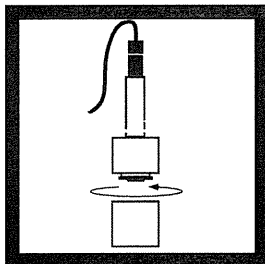
26. Holding the cup firmly with one hand, carefully insert the electrode holder into the sample cup and twist slightly to set the O-ring.



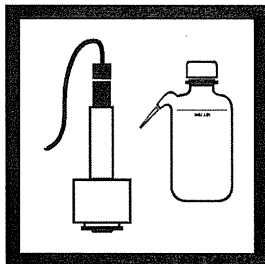
27. Turn the stirrer on and stir for 2 minutes.



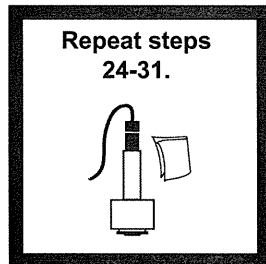
28. The display will show **Stabilizing...** until the reading is stable. Record or store the value.



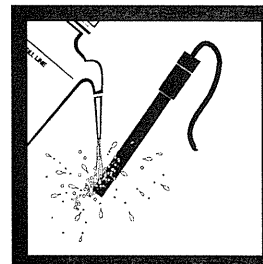
29. Remove the electrode holder assembly by holding the sample in one hand on a horizontal surface and pulling out with a slight twist. Shake any excess sample off the holder while keeping the holder in a vertical position.



30. Rinse the electrode holder with deionized water, including the inside sample reservoir via the reservoir vent. Be careful not to rinse oils, sludge or surfactants back into the air gap where the membrane is (it helps to retract the electrode from the electrode holder before rinsing).



31. Repeat steps 24-31 for each sample.



32. After the last sample, gently wash and blot the entire electrode. Turn the stirrer off.

AMMONIA IN SOLUTIONS CONTAINING FATS, FATTY ACIDS, OILS, AND SURFACTANTS, continued

Sampling and Storage

Collect samples in glass or polyethylene containers of convenient size. Clean new bottles by washing with deionized or distilled water. Fill the sample bottle completely and stopper immediately. Analyze the sample as soon as possible. Ammonia may be lost from samples at temperatures above 50 °C, so it is important to collect samples at less than 40 °C, or use a cooling coil between the bottle and sampling point if necessary.

Use sodium thiosulfate to destroy any residual chlorine immediately after collection so it will not react with ammonia in the sample.

If prompt analysis is not possible, preserve the sample with 0.8 mL of concentrated sulfuric acid per liter. Use a pH meter to be sure the pH of the preserved sample is between 1.5 and 2. Some wastewater samples may require more sulfuric acid to achieve this pH. Store the sample at 4 °C.

Do not use mercuric chloride (HgCl_2) as a preservative as ammonia complexes with mercuric ions. Before analysis, neutralize the sample to pH 7 with 1 N sodium hydroxide.

Accuracy Check

Note: *Hach standard concentrations are given as elemental nitrogen (N), not as NH_3 or NH_4^+ .*

Checking Electrode Response

To verify electrode response, measure the electrode potential (in mV) of two Ammonia nitrogen standard solutions, one decade apart in concentration, that bracket the expected sample range. For example, use 10 and 100 mg/L ammonia nitrogen standards to bracket an expected sample concentration of 30 mg/L. The two standards should have potentials -57 ± 3 mV apart at 25 °C.

Checking Calibration Accuracy

To verify calibration accuracy, measure the concentration of a known standard within the calibration range.

AMMONIA IN SOLUTIONS CONTAINING FATS, FATTY ACIDS, OILS, AND SURFACTANTS, continued

REQUIRED REAGENTS

Description	Quantity Required		Cat. No.
	Per Test	Unit	
Ammonia Electrode Filling Solution.....	2 drops	59 mL.....	44472-26
Ammonia Electrode Storage Solution	20 mL.....	500 mL.....	25412-49
Ammonia Ionic Strength Adjustor Pillows.....	varies	100/pkg.....	44471-69
Ammonia (Nitrogen) Standard Solutions:			
1 mg/L NH ₃ -N.....	20 mL.....	500 mL.....	1891-49
10 mg/L NH ₃ -N.....	20 mL.....	500 mL.....	153-49
100 mg/L NH ₃ -N.....	20 mL.....	500 mL.....	24065-49
Water, deionized.....	100 mL.....	4 L.....	272-56

REQUIRED APPARATUS

Air Gap Accessory Kit.....	1	each.....	50253-00
Ammonia Electrode, BNC.....	1	each.....	50250-00
Bottle, wash, 500 mL	1	each.....	620-11
<i>sensio</i> TM 4 Laboratory pH/ISE Meter	1	each.....	51775-00

Select one based on available voltage:

Stirrer, electromagnetic, 115 V, with stand and stir bar	1	each.....	45300-01
Stirrer, electromagnetic, 230 V, with stand and stir bar	1	each.....	45300-02

OPTIONAL REAGENTS AND APPARATUS

Ammonia Electrode Membrane Modules.....	3/pkg.....	50251-00
Cylinder, graduated, 25 mL	each.....	508-40
Electrode Washer	each.....	27047-00
Sulfuric Acid, concentrated	500 mL.....	979-49
<i>sensio</i> TM 2 Portable pH/ISE Meter.....	each.....	51725-00
TenSette [®] Pipet, 0.1-1.0 mL	each.....	19700-01
Pipet Tips, for TenSette [®] Pipet 19700-01.....	50/pkg.....	21856-96