

Platinum Series Combination Sodium Electrode

MANUAL

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SPECIFICATIONS

Specifications are subject to change without notice.

Cable Connector

BNC

Dimensions

Tip Diameter: 12 mm Tip Length: 115 mm

Total Length: 240 mm with dispenser

Cable Length: 1.0 m (36 in.)

Electrode Resistance

<1400 Mohms @ 30 °C (new)

Electrode Type

Combination with Ag/AgCl reference

Linear Region

1 mg/L to saturation

Minimum Sample Volume

25 mL

Range

0.010 mg/L Na⁺ to saturated solution Sample pH can range from 4 to 14

Response Time

Below 1.0 mg/L, 5–10 minutes Above 1.0 mg/L, 1–2 minutes

Slope

58 ±3 mV @ 25 °C

Storage Solution

1 N NaCl Sodium Electrode Storage Solution

Temperature Range

Routine Use: 0 to 50 °C Occasional Use: 0 to 80 °C Storage: -40 to 60 °C

SAFETY PRECAUTIONS

Please read this entire manual before unpacking, setting up, or operating this instrument. Pay particular attention to all danger and caution statements. Failure to do so could result in serious injury to the operator or damage to the equipment.

To ensure the protection provided by this equipment is not impaired, do not use or install this equipment in any manner other than that which is specified in this manual.

Use of Hazard Information

If multiple hazards exist, this manual will use the signal word (Danger, Caution, Note) corresponding to the greatest hazard.

DANGER

Indicates a potentially or imminently hazardous situation which, if not avoided, could result in death or serious injury.

CAUTION

Indicates a potentially hazardous situation that may result in minor or moderate injury.

NOTE

Information that requires special emphasis.

Precautionary Labels

Read all labels and tags attached to the instrument. Personal injury or damage to the instrument could occur if not observed.

This symbol, if noted on the instrument, references the instruction manual for operational and/or safety information.

SECTION 1 GETTING STARTED

1.1 Electrode Description

The Platinum Series Combination Sodium Electrode (*Figure 1*) is a reliable electrode designed for general purpose sodium measurement in all kinds of sample. Its renewable, free flowing reference junction provides fast and accurate sodium measurements. Diluted electrolyte and contaminants are removed from the junction after each pump to ensure stable, reproducible reference potentials.

The electrode uses an improved sodium-selective glass membrane containing an inner filling solution with a constant sodium ion concentration and glycerol as an antifreeze agent. The glass is a LAS (lithium aluminosilicate) glass. Special doping agents in the glass improve selectivity for sodium ions. A potential develops across the membrane that is proportional to the sodium concentration in the standard or sample.

Figure 1 Platinum Series Combination Sodium Electrode, Model Number 51925-00



The ideal sodium ion selective electrode responds (has a potential across the glass membrane) only to sodium ions. However, sodium electrodes are not perfectly selective and respond to other ions such as silver, hydrogen, lithium, ammonium, and potassium. The industry standard sodium glass electrode is made of NAS 11-18 glass that is 500–2000 times more sensitive to sodium ions than to potassium ions. The Hach glass has a selectivity coefficient of greater than 20,000 for sodium over potassium. Linearity, response speed, and conditioning time are also improved.

The Sodium Combination Electrode incorporates a glass sodium half-cell and a reference half-cell. The reference half-cell uses a silver–silver chloride reference element in a specially designed free-diffusion junction. The reference element is encased in Teflon tubing.

Electrolyte flows out through the open end of the reference element and into the sample solution without restriction. Traditional porous junctions often become clogged by precipitated silver chloride. This can cause large and unknown junction potentials that result in measurement errors. Hach's patented free-flowing reference system produces fast, stable potentials.

1.2 Electrolyte Description

The Ammonium Chloride electrolyte is formulated as a medium-viscosity gel that acts as a restriction device by limiting the release of electrolyte into the sample. Fresh reference electrolyte is supplied from a replaceable cartridge inside the electrode body by depressing the dispenser button. By refreshing the electrolyte at the reference junction, Hach's patented reference system eliminates junction potential errors caused by clogged frits. The gelling agent is non-ionic and does not interfere with the free flow of ions across the reference junction. Proper use and care of the reference half cell extends its life.

1.3 Preparing the Electrode for Use

1.3.1 Combination Electrode Components

General assembly procedures for the Platinum Series Combination Sodium Electrode follow. The electrode combines both the sensing half-cell and the reference half-cell in one electrode body. The reference electrode is a double junction type and uses an Ammonium Chloride Reference Electrolyte Gel.

The Platinum Series Combination Sodium electrode consists of these components (see *Figure 2*), from top to bottom:

Push-Button Dispenser

When the user pushes the black dispenser button down, 6 microliters of gel dispense from the bottom of the electrode, renewing the reference. When the gel is dispensed, the dispenser makes an audible "click". To prime the electrode quickly, press the dispenser all the way in and turn clockwise.

Electrolyte Cartridge

The cartridge contains enough gelled electrolyte for a minimum of 250 dispensations of gel. The clear body and black piston allow the user to see how much gel remains in the cartridge.

Electrode Body

The electrode has several unique features. It uses the same free-flowing reference technology as the standard Platinum Series electrode. The electrolyte cartridge is attached to the top of the electrode using a standard Luer taper fitting. For cleaning, a standard laboratory syringe attached to the electrode flushes the entire system.

Removable Bulb Guard

Leave the tip on for normal use or remove for electrode tip cleaning or when used in small test tubes. When possible, leave it on to protect the bulb.

Figure 2 **Electrode Components**



Preparing the Reference Half Cell 1.3.2

- 1. Remove the cap from the electrolyte cartridge and fit the cartridge outlet tube firmly onto the inlet tube of the electrode body.
- 2. Place the dispenser unit over the electrolyte cartridge. Screw the dispenser unit onto the electrode body until reaching the stop. **Do not over tighten.** The electrode will be automatically primed by screwing the dispenser unit onto the electrode body.
- 3. If electrolyte gel is not visible at the reference outlet, depress the pump button until it clicks; release the button. Repeat this procedure until gel is visible at the reference outlet. Alternatively, fully depress the button and rotate it clockwise until gel is visible at the reference outlet (1 to 3 rotations).

- **4.** Rinse the electrode with deionized water and blot dry with a paper towel. **Do not scrub the bulb.**
- **5.** Connect the BNC connector on the electrode to the BNC port on the meter.

Place the electrode into Sodium Electrode Storage Solution and condition overnight. Measure the sodium concentration according to the meter instruction manual.

1.3.3 Preparing the Sensing Bulb

The sensing half-cell is packaged and shipped dry from the factory with a dry cap over the glass membrane tip. Remove the cap and place the tip in Sodium Electrode Storage Solution or in 1 M sodium chloride solution and soak overnight. **Never let the glass dry out.** Always keep the electrode conditioned in solution. See *Section 1.3.1 Combination Electrode Components*.

1.4 Conditioning the Electrode

When an electrode bulb is immersed in an aqueous solution, a hydrated layer slowly forms at the bulb/liquid interface. The hydrated layer is chemically similar to the sample being measured, especially in samples of low ionic strength. The formation characteristics of this layer depend upon the type of bulb, the age of the bulb, the previous usage history of the bulb, the temperature and ionic strength of the aqueous solution, etc. This hydrated layer affects the sensing properties of the bulb, charge transfer, and ion transport. A dry bulb simply will not function properly.

Combination sodium electrodes with frits for reference junctions can suffer problems during storage and conditioning because storage solution trapped in the frit causes reference junction potentials to fluctuate. Hach's patented free-flowing reference junction eliminates these fluctuations, resulting in an accurate, and easy-to-use electrode when conditioning procedures are followed.

1.4.1 Normal Conditioning

(For routine measurements in samples 1 mg/L Na⁺ or greater):

Initial use: The sensing half-cell is packaged and shipped dry from the factory with a dry cap over the glass membrane tip. Remove the cap and place the tip in Sodium Electrode Storage Solution, or in 1 M sodium chloride solution and soak overnight. Install the electrolyte cartridge as described in *Section 1.3.2*. **Never allow the electrode to dry out!**

Between uses: Between measurements of samples with concentrations of 1 mg/L or greater, store the electrode in Sodium Electrode Storage Solution, or in 1 M sodium chloride solution. The solution keeps the bulb hydrated and prevents the reference gel from solidifying in the reference junction.

1.4.2 Low Ionic Strength Conditioning (For measurements in samples containing less than 1.0 mg/L Na⁺):

Initial use: The sensing half-cell is packaged and shipped dry from the factory with a dry cap over the glass membrane tip. Remove the cap and install the gel cartridge. Click the reference electrolyte dispenser until gel emerges from the tip. Rinse the electrode with a small stream of sample delivered through a disposable plastic Pasteur pipette, or with deionized water from a wash bottle. Place the tip in Sodium Electrode Storage Solution, or in 1 M sodium chloride solution and soak for at least one hour. Then condition in 0.10 mg/L sodium solution for at least 8 hours before calibrating using the Low Range Sodium Method on page 27.

Between uses: Between uses, in intervals of up to a few hours, the electrode can be stored in the sample (if not an extreme pH), or in a neutral low-ionic-strength solution such as tap water. Before measuring a new sample, refresh the reference electrolyte gel by clicking the dispenser several times. Carefully rinse the electrode to prevent contaminating the sample.

1.5 Checking the Sodium Electrode

Make a quick check of probe response by verifying proper electrode slope. Place the probe in a sodium solution, then increase the sodium concentration by a factor of ten. A one decade increase should increase the potential by 58 ± 3 mV.

Proper operation is also verified by a calibration curve with a slope of 58 ± 3 mV/decade. Normally, a calibration curve is the first step in a series of analyses.

- 1. Add 50 mL of deionized water to a plastic beaker. Add the contents of two Sodium/Potassium Ionic Strength Adjustor (ISA) powder pillows or 0.80 g of Sodium/Potassium ISA powder and stir to dissolve. If a magnetic stirrer is available, add a stir bar and place the beaker on the magnetic stirrer. Begin stirring at a moderate rate.
- **2.** Use a TenSette® Pipet or volumetric pipet to add 0.5 mL of a sodium standard such as 1000 mg/L or 100 mg/L.
- **3.** Place the sodium electrode in the sample. Record the potential when the reading stabilizes.
- **4.** Add 5.0 mL of the same sodium standard to the beaker and mix. When the potential stabilizes, record the reading.

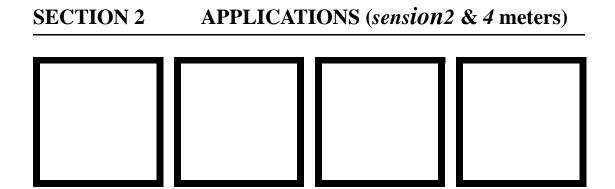
The potential should change by 58 ± 3 mV (at 25 °C). If the potential increase was less than 50 mV, clean the electrode glass membrane and re-test. Refer to *Section 4.2* on page 58. If the low slope remains, see *Section 6.1* on page 65.

1.6 Measuring Hints

These suggestions will improve the accuracy of your calibration and sample measurement.

- Always keep the electrode moist in 1 M NaCl or Sodium Electrode Storage Solution.
- Dispense electrolyte if reading becomes unstable, erratic, or if stabilization takes too long. An unstable reading may also indicate an air bubble in the reference line. Remove the electrode from the sample and invert it to view the reference junction. Depress the dispenser button repeatedly until the bubble is expelled (5 to 10 clicks should be sufficient). Rinse with sample and blot to dry. Immerse the electrode in the sample.

- All samples and standards should be at the same temperature (within 1 °C). If using a calibration curve, the standard should have been prepared at the same temperature as the samples.
- Adding Sodium Ionic Strength Adjustor (ISA) to each standard and sample gives a constant ionic strength; see *Section 5.4* on page *61*. Sodium ISA in powder form is available as unit-dose powder pillows or as bulk powder. An ISA solution could also be prepared.
- Rinse the electrodes with deionized water or a portion of the next solution to be measured. Blot dry with a paper towel between transfers. Do not rub the membrane. Rubbing can cause premature failure of the membrane.
- To achieve optimum accuracy, especially at low levels when extended sample stirring may occur, an insulating pad of cardboard or foam placed between the sample and the magnetic stirrer reduces heat transfer to the sample. No heat transfer will occur if the Hach Electromagnetic Stirrer is used. Use a magnetic stirrer and stir bar stirring at a constant, moderately fast rate to speed response and improve accuracy.
- Allow the potential to stabilize completely (< 1 mV/minute drift) before accepting a calibration point or sample reading. Meters set to higher resolutions will take longer to stabilize than meters set to lower resolutions.
- A multi-point calibration may ensure more accurate measurements than a two-point calibration.



Note: To measure sodicity in irrigation water, use the Salinity Appraisal Laboratory.

ANALYSIS PROCEDURE



1. Install the Ammonium Chloride Electrolyte Gel Cartridge in the Platinum Series Laboratory Sodium Electrode.

Note: The Hach sens**iOn™2** or **4** Meter may be used for this analysis.

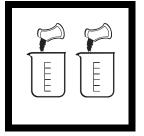


2. Connect the combination sodium electrode to the meter.

Note: Ensure the electrode has been conditioned for at least one hour in sodium electrode storage solution before use. See Section 1.3.2 on page 12.



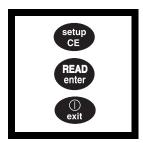
3. Prime the electrode by pushing the dispenser button until gel comes out of the reference junction. Rinse excess gel from the tip and the outlet.



Method 8357

4. In 50-mL beakers, prepare two 25-mL standard solutions of 100 and 1000 mg/L Na⁺. Add the contents of one Sodium Ionic Strength Adjustment (ISA) Buffer powder pillow to each standard. Stir to dissolve.

continued



5. Turn on the meter by pressing **I/O**. Press **SETUP**. Press **ENTER** until **BNC** appears. Press **EXIT**.

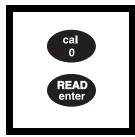
Note: To measure pH with a 5-pin connector, change the setting to 5-pin.



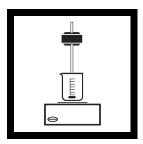
6. Press the **ISE mV** key until the display shows concentration units.

Note: A concentration reading may be displayed. Disregard and proceed.

Note: Temperature variation causes inaccurate measurements. Calibration and sample measurements should be made at the same temperature, ±1 °C.



7. Press CAL. The display will show CAL, ?, concentration units and the active keys. The units will be flashing. Use the arrow keys to select mg/L and press ENTER.



8. Add a stir bar to each cup. Place the beaker containing the 100 mg/L standard solution on an electromagnetic stirrer and stir at a moderate rate.

Note: Starting with the lowest concentration of standard reduces carry-over contamination and gives optimal probe response.

continued



9. Place the electrode into the beaker on the stirrer.



10. The display will show: Standard 1? and the value from the previous calibration. Press ENTER to accept the numerical value or use the number pad to edit the display to match the concentration of the 100 mg/L standard and press ENTER.

The display will show **Stabilizing...** until the reading is stable.

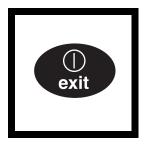


11. The display will show: **Standard 2?**. Remove the electrode from the beaker, rinse and blot dry.



12. Repeat *steps 8-10* for the 1000 mg/L standard.

continued



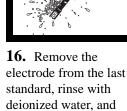
13. After the last calibration point is entered and the display reads **Standard 3?**, press **EXIT**.



14. Cal and Store? will be displayed. Press ENTER to accept the calibration or EXIT to leave the calibration mode without storing the calibration values.



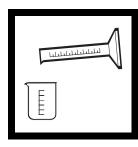
15. Press REVIEW. Use the UP ARROW key to scroll to the slope value. The slope should be 58 ±3 mV/decade. Press EXIT to return to measurement mode.



blot dry with a

paper towel.

Note: If the slope does not approximate this value, recalibrate. If the slope remains incorrect after repeating the calibration, see SECTION 6 TROUBLESHOOTING on page 65.



17. Accurately measure 25 mL of sample into a clean 50-mL beaker.

Note: For optimal accuracy, the sample should be the same temperature as the standards, ±1 °C.



18. Add the contents of one Sodium Ionic Strength Adjustor (ISA) powder pillow or 0.4 g of ISA powder to the beaker. Stir to dissolve.



19. Add a stir bar to the sample. Place the sample on a stirrer and stir at a moderate rate. Place the electrode in the sample.



20. The display will show **Stabilizing...** until reading is stable. Record or store this value.

continued



21. Remove the electrode from the sample after reading. Rinse the electrode. Repeat *steps 17-21* for each sample.

Note: Store the electrode in a sodium standard of similar concentration to samples to be analyzed during the next use. To store longer than one month, see Section 4.1 on page 57.

Sampling and Storage

Collect samples in an acid-cleaned glass or plastic container. Adjust the pH to 2 with nitric acid (about 2 mL per liter). Store preserved samples at room temperature up to six months. Adjust the pH to 9–10 with 8.0 M potassium hydroxide before analysis. Use pH Test Strips to measure pH. Do not use a pH electrode because it can cause silver contamination. Correct the test result for volume additions.

Accuracy Check

Checking Electrode Response

To verify electrode response, measure the electrode potential (in mV) of two Sodium Standard Solutions one decade apart in concentration, bracketing the expected sample concentration. For example, use 10 and 100 mg/L Sodium Standard Solutions to

continued

bracket an expected sample concentration of 30 mg/L. The two solutions should have potentials (in mV) that are 58 ± 3 mV apart at 25 °C. Both solutions must be above 1 mg/L Na⁺.

Checking Calibration Accuracy

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

Checking the Accuracy of the Sample Reading

To verify sample measurement accuracy, add a spike of Standard Sodium Solution with a TenSetteTM or volumetric pipet. Use the following table and formulas to calculate the percent recovery.

Measured Sample Concentration	Volume & Concentration of Standard to Add		
	V	С	CXV
1-2 mg/L	0.5 mL of	100 mg/L	50
3-6 mg/L	1.0 mL of	100 mg/L	100
7-15 mg/L	0.3 mL of	1000 mg/L	300
15-30 mg/L	0.5 mL of	1000 mg/L	500
30-60 mg/L	1.0 mL of	1000 mg/L	1000

continued

Percent Recovery

Use this formula to calculate the percent recovery when the sample volume is 25 mL.

M = calculated mass of sodium present after the spike (micrograms)

S = mg/L of Na^+ in sample (before spike)

C = concentration of standard used for spiking (mg/L)

V = spike volume (mL)

E = expected concentration after spiking (mg/L)

R = percent recovery (should be 95-100%)

A = actual reading on meter after spike (mg/L Na⁺)

$$M~=~S\times 25 + C\times ~V^*$$

$$E \; = \; \frac{M}{25 + V^*}$$

$$R \,=\, \frac{A}{E} \times\, 100\%$$

Method Performance

Precision

In a single lab using a standard solution of 25.0 mg/L and two electrodes with a single *sension2*, a single operator obtained a standard deviation of 0.65 mg/L sodium. Each electrode was exposed to seven test solutions with no rinsing in between and with a default stability of 0.5 mV/min.

^{*}Use these numbers from the table

continued

Interferences

Silver is a major interference. Low pH can interfere, but the ISA adjusts the pH to greater than 9.0, so it is eliminated. Potassium and ammonium interfere slightly.

REQUIRED REAGENTS	
Description Unit	Cat No.
Ammonium Chloride Reference Electrolyte Gel Cartridge	
Sodium Ionic Strength Adjustor (ISA), powder pillows 100/pkg	44515-69
Sodium Standard Solutions	
100 mg/L	23181-53
1000 mg/L500 mL	14749-49
Water, deionized	272-56
DECLUDED A DDA DA MUIG	
REQUIRED APPARATUS	1000 41
Beaker, 50 mL, polypropylene each each	
Bottle, wash, 500 mL	
Combination Sodium Electrode, Platinum Series, BNC connector each	
Cylinder, graduated, 25 mL, poly each	
sens ion TM 2 Portable pH/ISE Meter	51725-00
OR .	
sension TM 4 Laboratory pH/ISE Meter	
Stir Bar, 22.2 x 4.8 cm (⁷ / ₈ x ³ / ₁₆ '')	45315-00
Select one based on available voltage:	
Stirrer, electromagnetic, 115 V, with stand and stir bar	
Stirrer, electromagnetic, 230 V, with stand and stir bar	45300-02
OPTIONAL REAGENTS	
	202.22
Potassium Hydroxide Standard Solution, 8 N	
Sodium Ionic Strength Adjustor, bulk powder	
Sulfuric Acid, ACS 500 mL	979-49
OPTIONAL APPARATUS	
Pipet, TenSette®, 0.1 to 1.0 mL	19700-01
Pipet Tips, for 19700-01 TenSette Pipet 50/pkg	
Scoop, measuring, 0.5 gram.	
Scoop, measuring, 0.2 grams	

LOW RANGE SODIUM IN PURE WATER

 $(10 \text{ to } 3000 \, \mu\text{g/L Na}^+)$

This low range sodium test requires special conditioning procedures, *potassium chloride* electrolyte, and accurate temperature measurements. It is important to thoroughly read the procedure and accompanying notes as well as the *Sampling and Storage* section before proceeding. Complete the 9-hour conditioning step before the calibration step (see *Section 1.4.2* on page *14*).

Note: This test is for low range analysis only. If the electrode is conditioned to a low sodium concentration (as in this procedure), placing it in a concentrated Na+ sample (10 mg/L or higher) will swamp the electrode with sodium ion. Should this occur, repeat the overnight conditioning step described in Section 1.4.2 on page 14.

Notes

Prior to performing the test, prepare a 10 mg/L Na⁺ standard. Use a Class A pipet to dispense 10 mL of 1000 mg/L Na⁺ standard into a 1.0 L volumetric flask. Dilute to the mark with deionized water. This is a 10 mg/L Na⁺ standard.

Be sure to measure millivolt potentials of all sodium standards at the same temperature ± 0.5 °C. Also, the sample and standards must be measured at the same temperature, ± 1 °C. This procedure keeps temperature error to a minimum by using a spiked additions method of calibration. Even so, the analyst should use room temperature 100 mg/L Na^+ standard.

One recommendation is to use a temperature bath slightly above room temperature (25 °C) to equilibrate the standard temperature and sample temperature before measuring mV potentials. Use a laboratory grade thermometer to monitor the temperature. A one degree centigrade difference may result in as much as a 0.4 mV inaccuracy. This temperature variation will, in turn, decrease accuracy of concentration measurements.

continued

ANALYSIS PROCEDURE



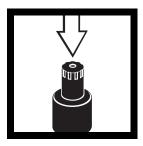
1. Install the Potassium 2. Connect the Chloride Electrolyte Gel Cartridge in the Platinum Series **Combination Sodium** Electrode.

Note: Only the sension TM4 Meter may be used for this analysis.



combination sodium electrode to the meter.

Note: Ensure the electrode has been conditioned for at least 8 hours in Sodium Electrode Storage Solution before its initial use.



3. Prime the electrode by pushing the dispenser button until gel comes out of the reference junction. Rinse excess gel from the tip and the outlet.





4. Condition the electrode in sodium electrode storage solution for a minimum of 1 hour before use. Then condition in 0.10 mg/L sodium for at least 8 hours.

Note: To make 100 mL of 0.10 mg/L Na+ standard, use a TenSette™ Pipet to deliver 0.10 mL of 100 mg/L Na+ into a 100-mL volumetric flask and dilute to the mark. Mix well.

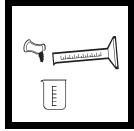
LOW RANGE SODIUM IN PURE WATER (10 to 3000 $\mu g/L$ Na⁺),

continued



5. Accurately measure 400 mL of deionized water into a plastic 500-mL graduated cylinder.

Note: The deionized water must be at room temperature. Temperature variation causes inaccurate measurements. Calibration and sample measurements should be taken at the same temperature ±0.5 °C.



6. Pour the water in the cylinder into a 600-mL plastic beaker. Add the contents of one Sodium Ionic Strength Adjustor powder pillow to the solution.

Add a large stir bar (50.8 x 7.9 mm) to the beaker. Place the beaker on an electromagnetic stirrer and begin stirring at a moderate rate.

Note: Stirring at a constant, moderately fast rate speeds response and improves accuracy. For very accurate work, especially at low levels when extended stirring may occur, use the electromagnetic stirrer listed under REQUIRED APPARATUS. Using any other stirrer requires an insulating pad to minimize heat transfer to the sample.



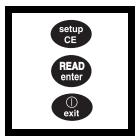
7. Remove the electrode from the 0.10 mg/L sodium standard, dispense gel, and rinse excess gel away with deionized water. Place the electrode in the 600-mL beaker, submerging the tip below the solution surface.



8. Using a TenSette[®] Pipet, add 0.4 mL of 10 mg/L Sodium Standard Solution to the solution in the beaker. (This makes 400 mL of 0.010 mg/L or 10 μg/L sodium.)

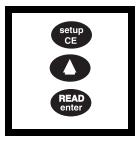
Allow the electrode to condition for 15 minutes in this solution before proceeding.

continued



9. Turn on the meter by pressing I/O. Press SETUP. Press ENTER until BNC appears. Press EXIT.

Note: To measure pH with a 5-pin connector, change the setting to 5-pin.



10. Press **SETUP** and use the **UP ARROW** key to scroll to **Stabilizing**. Press **ENTER**. Use the keypad to edit the display to 0.1 mV/min. Press **ENTER** to accept the value. Press **EXIT**.



11. Press **ISE mV** until **mV** is displayed. The mV potential should be displayed.



12. When the display stabilizes (**Stabilizing...** no longer appears in the display) record the potential and the temperature of the standard.

If the electrode is conditioned properly, the mV potential should not change more than 0.1 mV every minute. If the potential drifts one direction more than 0.1 mV every minute, leave the electrode in the 600-mL beaker until the drift has slowed.

If the electrode drifts 10 mV or more in the positive direction (for example, from -175 to -165 mV), repeat *steps 5-11*, except do not dispense electrolyte gel again as directed in *step 7*.

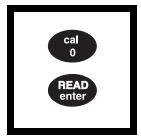
Note: Use a lab grade thermometer to measure the temperature to 0.5 °C.

continued



13. Press **ISE mV** to toggle to concentration units.

Note: A concentration reading may be displayed. Disregard and proceed.



14. Press the **CAL** key. **CAL?** will be displayed. Scroll to µg/L and press **ENTER.**

The display will show: **Standard 1?**



15. Use the number pad to edit the display to match the concentration of solution in the 600 mL beaker (10 μg/L). This is the concentration of *step 1* in the Low Level Sodium Calibration table below. Press **ENTER**. The display will show **Stabilizing...**, then **Standard 2?**.

Note: The pH/ISE meter will select a stable reading according to the 0.1 mV/min signal stability parameter entered in this procedure. This reading is stable when Stabilizing... no longer appears in the display.



16. Pipet the corresponding additional volume of 10 mg/L Na⁺ standard in *Table 1*, (*step 2*, 0.4 mL of 10 mg/L Na⁺). Wait the time specified in *Table 1* to allow the membrane to respond. Enter the concentration (in μg/L) in *Table 1*, (*step 2*, 20 μg/L) by using the number pad to edit the display, and then pressing **ENTER**.

continued



17. Repeat *step 16* by adding the additional volumes of 10 and 100 mg/L Na⁺ standard listed in *Table 1* until all seven standards have been read by the meter and entered. The meter will display **Store?**. Press **ENTER** to store the calibration and return to reading mode.

Note: If an error message is displayed see TROUBLESHOOTING, on page 65.

Note: To review your calibration points, press the REVIEW key. Scroll with the UP arrow to obtain the mV potential and concentration of each point entered.



18. Remove the electrode from the last standard, rinse well with deionized water, and blot dry.

Note: Save the solution in the 600-mL beaker with the 2.00 mg/L Na+ for later calibration checks.



19. Accurately measure 400 mL of sample into a 500-mL graduated cylinder.

Note: The sample must be at the same temperature as the standard solution in the 600-mL beaker used to perform the calibration.



20. Pour this 400 mL of sample into a 600-mL beaker. Add a magnetic stir bar, place the beaker on an electromagnetic stirrer. Stir at a moderate rate.

Note: If the electrode is placed in a sample of high sodium concentration, errors may result and reconditioning to lower sodium levels will be required.

Table 1

Step	Vol. 10 mg/L	Vol. 100 mg/L Na+ Standard Added	Concentration	
Step	Na+ Standard Added		μ g/L	Time
1	0.40 mL		10	until mV stabilizes
2	0.40 mL		20	10 min.
3	1.2 mL		50	10 min.
4		0.2 mL	100	5 min.
5		0.4 mL	200	5 min.
6		1.2 mL	495	5 min.
7		1.5 mL	1977	5 min.

continued



21. Add the contents of one Sodium Ionic Strength Adjustor Powder Pillow.



22. Place the electrode into the sample.

Wait 10 minutes to allow electrode to condition to the low level sodium in solution.



23. Then read the sodium concentration from the display after it stabilizes (Stabilizing... no longer appears in the display). This is the sample concentration. Record this value.

Note: If the sodium concentration is outside the 10 to 2000 µg/L calibration range, press the ISE mV key. If the mV reading is greater than the mV at 2000 µg/L Na+, analyze the sample using the procedure for Potable, Ground, and Irrigation Water. If the mV reading is less than the mV reading of the 10 µg/L standard, there is less than 10 µg/L Na+ in the sample.

Sampling and Storage

Analyze immediately after sampling. If immediate analysis is impossible, cool samples to 4 °C and analyze within six hours.

Accuracy Check

Checking Electrode Response

To verify electrode response at these low levels of sodium, the millivolt potential should increase upon each addition of 100 mg/L Na⁺. Using the Low Level Sodium procedure, at least a 4.0 mV increase should be observed from $step\ 1$ to $step\ 2$ (10 µg/L to 20 µg/L Na⁺). Each additional spike should increase the mV reading substantially from the previous change. If this is not the case, check the purity of the standard used. If this is not the problem, the electrode is probably not conditioned for low sodium levels.

continued

Checking Calibration Accuracy

- 1. Fill the 500-mL graduated cylinder to the 400-mL mark with deionized water.
- 2. Pour this solution in a 600-mL beaker and add a stir bar.
- **3.** Pipet 0.2 mL of 100 mg/L Sodium Standard into the 600-mL beaker.
- **4.** Add the contents of one Sodium Ionic Strength Adjustor powder pillow and place on an electromagnetic stirrer.
- 5. Rinse the calibrated electrode before placing in solution. Measure the concentration of the solution. The reading should be approximately 50 µg/L.

Also, the beaker with the 2000 $\mu g/L$ Na⁺ standard used in the calibration may be used as a check on the calibration. It should read close to 2000 $\mu g/L$.

Checking Accuracy of Sample Measurement

To check the accuracy of the sample measurement, a spike of standard may be added to the sample and percent recovery calculated. *Table 2* shows volumes of 100 mg/L Na⁺ to be added for the proper spiking of samples. Following this is a calculation of percent recovery. For the Platinum Series Sodium Combination Electrode the calculated percent recoveries should be 90–100%.

Table 2 Spiking Volumes of 100 mg/L Standard

Measured Sample Conc. Na+ (μg/L)	Vol. of 100 ppm Std. added to sample	CXV
10 to 49	0.1 mL	10
50 to 99	0.2 mL	20
100 to 299	0.4 mL	40
300 to 599	1.2 mL	120
600 to 2000	2.0 mL	200
1000 to 2000	4.0 mL	400

continued

Percent Recovery

Calculate the percent recovery using the formulas below:

M = Calculated mass of sodium present in the sample after the spike (in micrograms)

S = Sample reading (in mg/L Na+)

C = Concentration of standard used to spike in mg/L

V = Volume of standard used in spike in mL

E = Expected concentration reading after addition of spike

A = Actual concentration reading after adding spike

R = Percent recovery (should be 90-100%)

$$M = S \times 400 + (C \times V)^*$$

$$E = \left[\frac{M}{400 + V^*} \right]$$

$$R = \frac{A}{F} \times 100\%$$

Method Performance

Precision

In a single lab using a standard solution of 25 μ g/L and two electrodes with a single *sension4*, a single operator obtained a standard deviation of 1.83 mg/L Na⁺. Each electrode was exposed to seven test solutions with no rinsing in between and with a default stability of 0.1 mV/min.

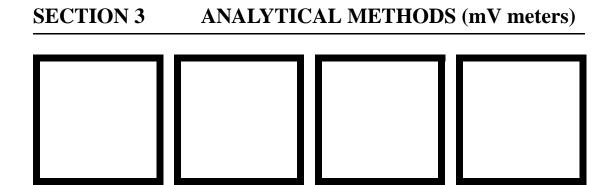
Interferences

The Sodium ISA is formulated to remove most interferences. Silver is a major interference.

^{*}Use the numbers from Table 2.

continued

REQUIRED REAGENTS	
Description Unit	Cat. No
Potassium Chloride Reference Electrolyte Gel Cartridge	25469-02
Sodium Ionic Strength Adjustor Powder Pillows 100/pkg	
Sodium Standard Solutions 100 mg/L as Na ⁺	23181-53
Sodium Standard Solutions 1000 mg/L as Na ⁺ 500 mL	14749-49
Water, deionized4L	272-56
DECLUDED ADDADATUC	
REQUIRED APPARATUS	620.11
Bottle, wash, 500 mL	
Combination Sodium Electrode, Platinum Series, BNC connector	
sension TM 4 Laboratory pH/ISE Meter	
Beaker, 600 mL, polypropylene	
Cylinder, graduated, 500 mL, polypropylene	
Pipet, TenSette®, 0.1 to 1.0 mL	
Pipet Tips, for TenSette Pipet	
Stir bar, 22.2 x 4.8 mm (⁷ / ₈ x ³ / ₁₆ ")	
Stir bar, 50.8 x 7.9 mm (1 ¹ / ₈ x ³ / ₁₀ ")	20953-55
Select one based on available voltage:	45200.01
Stirrer, electromagnetic, 115 V, with stand and stir bar	
Stirrer, electromagnetic, 230 V, with stand and stir bar	45300-02
OPTIONAL REAGENTS	
Sodium Ionic Strength Adjustor (ISA), powder454 g	44515-01
Water, deionized	
OPTIONAL APPARATUS	
Water Bath, circulating	26163-00
Flask, volumetric, poly, 50 mL	
Flask, volumetric, poly, 100 mL	



DIRECT MEASUREMENT METHOD BY GRAPHING

Most modern pH/mV/ISE meters, including the Hach *sension*TM2 and *sension4*, allow the user to perform calibrations for the species and concentration range of interest. Once calibrated, the meter will give a direct reading of the concentration for that species. Unlike many meters, which are limited to linear calibrations, the *sension2* and *sension4* meters can cover a larger concentration range that has both linear and non-linear electrode response regions. These *sension* meters will then accurately calculate the concentration from the electrode potential and display the concentration directly in user-selected units such as ppm, mg/L, M, etc.

When the user only has access to a pH/mV meter, the user can still obtain results with the use of semi-log graph paper, plotting concentration (on the Y log axis) vs. potential (in mV, on the X axis). Procedures for graphing these data are described below.

Examples of calibration curves are shown in *Figure 3* and *Figure 4*, below.

Figure 3 Typical Calibration Curve

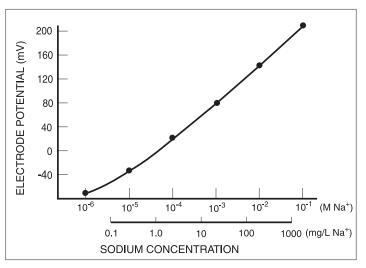
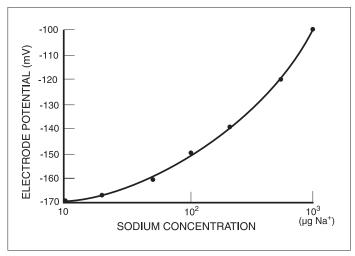


Figure 4 Low Level Calibration Curve



Required Apparatus and Solution for Direct Measurement Method

- Combination Sodium Electrode
- mV or mV/pH meter
- Magnetic stirrer and stir bars
- Plastic beakers, 50- or 100-mL
- Sodium Standard solutions, to cover range of interest
- Sodium ISA powder pillows, or Sodium ISA powder
- Ammonium Chloride Reference Electrolyte Gel Cartridge (if not using a Hach meter with built-in dispenser)
- Semilogarithmic Graph paper, two to five cycles (if not using an ISE meter capable of nonlinear calibrations)

Preparing Standards for Direct Measurement Method

The concentration and number of standards to prepare depends on the sample concentrations. If the samples all have similar Na^+ concentrations and are in the linear region of the probe (above $1~mg/L~Na^+$ or above $10^{-4}~M$), use a short direct method of two standards (one or two powers of ten, or decades, apart in

concentration) that bracket the expected sodium level. To cover a wider range of sodium concentrations, or to go below 1 mg/L (10-4 M), use multiple standards. See *Figure 3 and Figure 4*. Standard solutions can be purchased or prepared by serial dilution of a concentrated sodium solution. Hach standards of 1000 ppm, 100 ppm, and 0.1 M are available; see *Replacement Parts*.

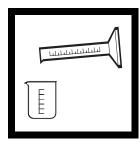
To prepare your own standards:

- 1. Measure 100 mL of the concentrated standard (e.g., 1000 mg/L Na⁺) into a 1-liter volumetric flask.
- 2. Fill to the mark with deionized water. Mix. This is a tenfold dilution of the original (e.g., 100 mg/L). Pour the solution into a 1-liter plastic container to store. Label the container.
- **3.** Repeat the dilution using the first tenfold dilution (e.g., 100 mg/L) to prepare another tenfold dilution (e.g., 10 mg/L).
- **4.** Repeat, diluting each dilution, to cover the lowest expected level (e.g., 0.01 mg/L Na⁺ or 10⁻⁶ M).

Notes

- A 100-mg/L sodium standard solution can be prepared by adding 0.2542 g sodium chloride (analytical reagent grade, oven-dried at 140 °C) to a 1000-mL volumetric flask and diluting to the mark with deionized water.
- The serial dilutions also could be done from a 0.1–M standard, producing 10⁻² M, 10⁻³ M, 10⁻⁴ M, and so forth.
- Sodium concentrations can be expressed in units of moles/L, or mg/L of sodium. For example, 2300 mg/L Na⁺ (sodium) is equivalent to 10⁻¹moles/L (M).

Preparing a Calibration Curve



1. Measure 25 mL of a standard solution in a graduated cylinder and pour into a 50- or 100-mL beaker.

Note: Select standards covering the range of interest. In the linear response region of the electrode, or to cover a wide concentration range, one standard per decade is usually used. For measurements in the nonlinear range, use standards which bracket the expected range. The more points used, the better the accuracy.



2. Add the contents of one Sodium ISA powder pillow, or add 0.4 g of Sodium ISA powder.



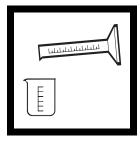
3. Add a magnetic stir bar, place the beaker on a magnetic stirrer and begin stirring at a moderate rate. Allow most of the powder to dissolve.



4. Place the electrode in the standard.



5. When a steady reading is obtained, record the potential.



6. Prepare the next standard solution as in *steps 1* and 2.



7. Remove the electrode from the previous solution, rinse with deionized water and blot dry with a paper towel.



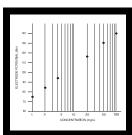
8. Immerse the electrode in the new solution.



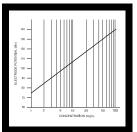
9. Record the potential when the reading is stable.

Repeat Steps1-9 for all Standards

10. Repeat steps 1–9 for all standards to be used for the calibration curve.



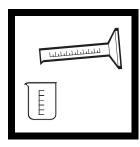
11. Plot the potentials vs. sodium concentrations on semilogarithmic graph paper. Concentration goes on the log axis and may be plotted as mg/L (ppm), moles/L, pNa⁺, or any convenient concentration unit. Potentials are plotted on the linear axis; see *Figure 5* on page 45.



12. Draw a best-fit line through the points. With concentrations above 1 mg/L $(7x10^{-5} \text{ M})$ there should be a straight line with a slope of $58 \pm 3 \text{ mV/decade}$ (at $25 \,^{\circ}\text{C}$).

Note: The best-fit line also can be determined by linear regression analysis, an option on many calculators and the Hach pH/ISE Meter. The regression analysis provides a correlation coefficient and the slope of the line. With these two factors, sample concentrations can be calculated from the measured potentials.

Procedure



1. Measure 25 mL of sample with a graduated cylinder. Pour it into a 50- or 100-mL beaker.



2. Add the contents of one sodium ISA powder pillow or 0.4 g of ISA powder.



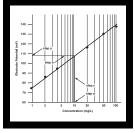
3. Add a magnetic stir bar, place the beaker on a magnetic stirrer and begin stirring at a moderate rate.



4. Remove the electrode from the previous solution, rinse with deionized water, or a portion of the next sample, and blot dry with a paper towel.



5. Place the electrode in the sample. Record the potential when a stable reading is obtained.



6. Determine the sodium concentration from the calibration curve. See the example below.

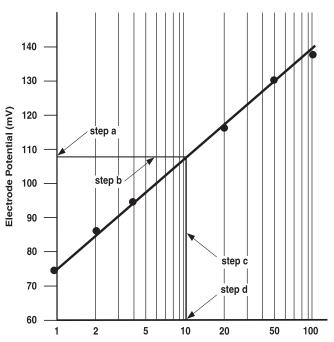
Example:

A water sample was measured with the sodium electrode using the above procedure. The electrode potential was 100 mV. What is the sodium concentration of the sample?

- **a.** Locate the sample potential on the linear axis.
- **b.** Draw a horizontal line from the potential on the linear axis to the calibration curve.
- **c.** From that point on the curve, draw a vertical line down to the concentration logarithmic axis.
- **d.** Read the point on the logarithmic axis as the sample concentration.

Figure 5 is an example of a calibration curve. The electrode potential is located on the linear axis (step a) 108 mV. A horizontal line is drawn to the calibration curve (step b). A vertical line is drawn down to the logarithmic axis (step c). The sample concentration is 15 mg/L sodium (step d).

Figure 5 Linear Calibration Curve

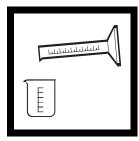


REQUIRED REAGENTS		
Description	Unit	
Ammonium Chloride Reference Electrolyte Gel Cartridge		
Sodium Ionic Strength Adjustor (ISA), powder pillows	100/pkg	44515-69
Sodium Standard Solutions		
100 mg/L	1000 mL	23181-53
1000 mg/L	500 mL	14749-49
Water, deionized	4.0 L	272-56
REQUIRED APPARATUS	_	
Beaker, 50 mL, polypropylene		
Bottle, wash, 500 mL		
Combination Sodium Electrode, Platinum Series, BNC		
Cylinder, graduated, 25 mL, poly		
sension TM I Portable pH/mV Meter		
Stir Bar, 22.2 x 4.8 cm (7/8 x 3/16")		45315-00
Select one based on available voltage:		
Stirrer, electromagnetic, 115 V, with stand and stir bar		45300-01
Stirrer, electromagnetic, 230 V, with stand and stir bar		45300-02
Semi-log Graph Paper, 2 cycle		23062-00
OPTIONAL REAGENTS		
Sodium Ionic Strength Adjustor, bulk powder	454 g	44515-01
OPTIONAL APPARATUS		
Cylinder, graduated, 25 mL, poly	each	1081-40
Pipet, TenSette [®] , 0.1 to 1.0 mL		
Pipet Tips, for 19700-01 TenSette Pipet		
Scoop, measuring, 0.5 gram		
Scoop, measuring, 0.2 grams		

LINEAR CALIBRATION WITH TWO STANDARDS

Linear calibration with two standards is a frequently used calibration method for direct measurement. The two-point calibration may be quickly and easily repeated during each series of measurements. The two standards are chosen in the expected concentration range of the samples. They often differ in concentration by one decade.

Note: The two-point calibration should be used only in the linear region. See Measuring Hints.



1. Measure 25 mL of standard 1. Pour into a beaker. Add one powder pillow or 0.4 g of Sodium ISA powder.



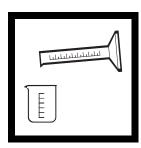
2. Stir. Place the electrode into the solution. Record the potential when it stabilizes.



3. Rinse and blot the electrode dry with a paper towel and repeat *steps 1* and 2 with standard 2.



4. Calculate the calibration slope using equation 1 below.



5. Determine the sample concentration by measuring 25 mL of sample into a beaker and adding one powder pillow or 0.4 g of Sodium ISA powder.



6. Rinse and blot the electrode dry with a paper towel. Place the electrode into the stirring sample.



7. Dispense electrolyte if necessary. Measure the potential.



8. Calculate the sample concentration using equation 2 below. Replace the electrolyte cartridge cap when the cartridge is removed from the electrode.

LINEAR CALIBRATION WITH TWO STANDARDS, continued

Equation 1

$$S = \frac{E_1 - E_2}{\log C_1 - \log C_2}$$

Where:

S = slope in mV/decade

 E_1 and E_2 = potentials of the standards

 C_1 and C_2 = the sodium concentrations of standards 1 and 2

Equation 2

$$\boldsymbol{C}_{x} \; = \; \boldsymbol{C}_{1} \times \; 10^{\Delta E/\;\; S}$$

$$\Delta E = E_x - E_1$$

Where:

 E_x = sample potential

E₁ = standard potential

 C_x = sample concentration

 C_1 = standard concentration

S = the electrode slope

Example

Standard 1 (C_1) = 10.0 mg/L and its potential (E_1) = 113.2 mV.

The slope is 58.1 mV/decade.

The sample potential $(E_x) = 136.9 \text{ mV}$

$$\Delta E = 136.9 - 113.2 = 23.7 \text{ mV}$$

$$C_{\rm x} = 10 \ {\rm mg/L} \ {\rm x} \ 10^{23.7/58.1} = 25.6 \ {\rm mg/L}$$

LINEAR CALIBRATION WITH TWO STANDARDS, continued

Periodically remeasure the standard and compare it with the calibration value. If the potential of the standard has changed, the offset potential has changed and a new calibration should be done.

If sample temperature and electrode slope are reproducible, the one-standard calibration may be used more often. The two-standard calibration is required when temperatures vary.

REQUIRED REAGENTS		
Description	Unit	Cat No.
Ammonium Chloride Reference Electrolyte Gel Cartridge	2/pkg	25958-02
Sodium Ionic Strength Adjustor (ISA), powder pillows	100/pkg	44515-69
Sodium Standard Solutions		
100 mg/L	1000 mL	23181-53
1000 mg/L	500 mL	14749-49
Water, deionized	4.0 L	272-56
REQUIRED APPARATUS		
Beaker, 50 mL, polypropylene	each	1080-41
Bottle, wash, 500 mL		
Combination Sodium Electrode, Platinum Series, BNC		
Cylinder, graduated, 25 mL, poly		
sension TM 1 Portable pH/mV Meter		
Stir Bar, 22.2 x 4.8 cm (7/8 x 3/16")		
Select one based on available voltage:		
Stirrer, electromagnetic, 115 V, with stand and stir bar		45300-01
Stirrer, electromagnetic, 230 V, with stand and stir bar		
OPTIONAL REAGENTS		
Sodium Ionic Strength Adjustor, bulk powder	454 g	44515-01
OPTIONAL APPARATUS		
Pipet, TenSette®, 0.1 to 1.0 mL		19700-01
Pipet Tips, for 19700-01 TenSette Pipet		
Scoop, measuring, 0.5 gram		907-00
Scoop, measuring, 0.2 grams		638-00

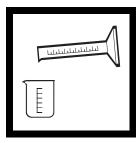
KNOWN ADDITION METHOD USING pH/mV METERS

In the known or standard addition method, a known amount of sodium solution is added to the sample and the original sodium level is calculated from the change in potential. This technique requires the electrode slope to be known and the measurements to be in the linear region. A calibration curve is useful to show the linear region of the probe and provide the slope. The *sension*TM4 provides a program that automatically calculates results with a standard addition method. The method described below is only necessary if a pH/mV meter is used.

This method may be used when only a few samples are to be analyzed. The known addition method may be used without a calibration curve.

Best results are obtained when the sodium level is roughly doubled by the standard addition. Therefore, the sample [Na⁺] should be roughly known, either from prior knowledge with the sample type or estimating it from the potential, using direct measurement (a calibration curve).

Procedure



1. Measure 50 mL of sample with a graduated cylinder. Pour into a beaker.



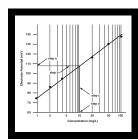
2. Add the contents of two powder pillows or 0.8 g of Sodium ISA, add a stir bar, place the beaker on a magnetic stirrer and begin stirring.



3. Place the electrode into the sample.



4. Record the mV potential when it stabilizes.



5. Use a calibration curve (see *Figure 5* on page *45*) to estimate the sample sodium concentration from the potential.



6. Using equation 1 and example 1, below, calculate the volume of sodium standard needed to roughly double the [Na⁺].



7. Add the calculated volume of standard into the stirring sample.

Note: Using fairly concentrated standards will change the total solution volume less because smaller volumes are added. Addition volumes of 1% or less of the sample volume are assumed in the calculation below.



8. Record the potential when it stabilizes. Calculate the sample concentration using equations 2 and 3 and example 2, below.

Equation 1:

$$V_{standard} = \frac{C_{sample} \times V_{sample}}{C_{standard}}$$

Where V = volume and C = concentration

Example 1

How many mL of a 100-mg/L sodium standard are needed when the electrode potential of a 50-mL sample suggests the approximate sodium concentration is 1 mg/L?

$$V_{standard} = \frac{1 \times 50}{100} = 0.50 \text{mL}$$

Add 0.5 mL of sodium standard to the sample.

Equations 2 and 3:

$$C_{Na+} = \frac{V_{standard} \times C_{standard}}{V_{sample} \times (10^{\Delta E/S} - 1)}$$

$$\Delta E = E_2 - E_1$$

Where:

 E_1 and E_2 = the first and second potentials measured

S =the electrode slope (a positive value)

 C_{Na}^{+} = the sodium concentration in the sample

Example 2

The sample potential is 87.6 mV. Upon adding 0.20 mL of 2300 mg/L (0.1 M) standard the potential goes to 110.6 mV.

$$\Delta E = 110.6 - 87.6 = 23.0 \text{ mV}$$

$$V_{\text{standard}} = 0.20 \text{ mL}; C_{\text{standard}} = 2300 \text{ mg/L}$$

$$V_{\text{sample}} = 50 \text{ mL}$$

Slope S was experimentally determined to be 56.6 mV/decade.

$$C_{\text{Na+}} = (0.20 \text{ x } 2300) \div [50 \text{ x } (10^{23.0/56.6} - 1)]$$

$$C_{\text{Na+}} = 5.90 \text{ mg/L}$$

REQUIRED REAGENTS		
Description	Unit	Cat No.
Ammonium Chloride Reference Electrolyte Gel Cartridge		
Sodium Ionic Strength Adjustor (ISA), powder pillows	100/pkg	44515-69
Sodium Standard Solutions		
100 mg/L	500 mL	23181-53
1000 mg/L	500 mL	14749-49
Water, deionized	3.78 L	272-56
DECLUDED A DDA DA MAIG		
REQUIRED APPARATUS		1000 41
Beaker, 50 mL, polypropylene		
Bottle, wash, 500 mL		
Combination Sodium Electrode, Platinum Series, BNC connec		
Cylinder, graduated, 25 mL, poly		
sension TM I Portable pH Meter		
sension TM 2 Portable pH/ISE Meter		
sension TM 3 Laboratory pH Meter		
Stir Bar, 22.2 x 4.8 cm (⁷ / ₈ x ³ / ₁₆ ")		45315-00
Select one based on available voltage:		
Stirrer, electromagnetic, 115 V, with stand and stir bar		
Stirrer, electromagnetic, 230 V, with stand and stir bar		45300-02
OPEIONAL DE ACENTEC		
OPTIONAL REAGENTS	454	44515.01
Sodium Ionic Strength Adjustor, bulk powder	454 g	44515-01
OPTIONAL APPARATUS		
Cylinder, graduated, 25 mL, poly	each	1081-40
Pipet, TenSette [®] , 0.1 to 1. 0 mL		19700-01
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg	21856-96
Scoop, measuring, 0.5 gram		
Scoop, measuring, 0.2 grams		638-00

SECTION 4 ELECTRODE MAINTENANCE

The Platinum Series Combination Sodium electrode contains a sensing bulb and a liquid reference junction optimized for performance in saline solutions. This sensitive instrument has been designed to give trouble-free use, but requires careful handling to extend longevity. This section explains methods for cleaning and storing the Platinum Series Combination Sodium electrode.

4.1 Storing the Electrode:

Proper electrode storage requires different approaches based on how long the electrode will be stored, how quickly the electrode needs to be brought to working condition, and the type of sample being measured. Storage affects the ammonium chloride electrolyte gel, the Ag/AgCl reference element, and the sensing bulb. Do not allow the electrode to dry out; this may permanently reduce performance.

Do not leave the electrode in solutions with low sodium concentrations for extended periods (especially deionized and ultrapure water). This can shorten electrode life.

Intermittent storage—Store the electrode in either Sodium Storage Solution (1 M sodium chloride), or a sodium standard solution in the range of samples being measured with ISA added. Before measuring a new sample, refresh the reference electrolyte gel by clicking the dispenser several times. Carefully rinse the electrode to prevent contaminating the sample.

Overnight storage and longer-term storage—When not used for up to one week, store the electrode in a small volume of Sodium Storage Solution (1 M sodium chloride), or a sodium standard solution with ISA added. Fill the electrode cap half full with standard solution and insert the electrode end into the cap for temporary storage or for field transportation. In the laboratory, store the electrode in a beaker of standard. Prior to use, click the electrolyte gel dispenser. A dilute gel/storage solution will initially be ejected from this tube. The reference gel of thicker viscosity will follow. Eliminating the dilute gel/storage solution will guarantee continuity of the reference junction with the sample. Blot the reference junction with a tissue to clearly see that the electrolyte gel, not gel diluted with storage solution, is emerging.

Shelf Storage—For very long-term storage, store the electrode in sodium electrode storage solution. If storing longer than two weeks, replace the electrolyte cartridge before using and reprime the reference electrode.

4.2 Cleaning the Electrode

A contaminated bulb or fouled electrode may cause slow response times. Clean the electrode only after conducting the steps described in *SECTION 6 TROUBLESHOOTING* or when the electrode is known to be dirty. According to the instructions below, clean the electrode according to the type of contaminant present:

General contamination—Immerse the electrode tip in 0.1 N Hydrochloric Acid followed by immersion in 0.1 N Sodium Hydroxide and again in 0.1 N Hydrochloric Acid, each for a 2-minute period. Rinse with deionized water and soak in deionized water for at least 15 minutes.

Oils and fats—Immerse the electrode tip in a detergent solution such as AlconoxTM. Use a soft brush or ultrasonic bath if necessary. Avoid scratching the bulb.

Organic films—Use an appropriate solvent, such as methanol or acetone.

After cleaning, refresh reference half-cells by pumping fresh electrolyte through them. If the electrode has been sitting in a sample for several weeks, the reference gel will become diluted. In this case, discard the cartridge and replace it with a fresh one.

If these steps fail to improve electrode response, replace the electrode.

5.1 Theory of Operation

The glass sensing membrane in the probe is in contact with an internal filling solution and the external sample solution. A constant potential is maintained inside the glass bulb due to the fixed ion concentrations of the internal filling. The outer, hydrated surface of the glass bulb can exchange sodium ions with the sample solution, setting up a potential which is proportional to the concentration of sodium ions in solution.

A reference electrode is required to complete the electrical circuit through the pH/mV meter, and establish a stable, reproducible potential. In the complete electrochemical cell consisting of the reference and indicator half cells, only the potential across the sensing membrane should vary.

The measured cell potential is related to the sodium ion concentration by the Nernst Equation:

 $E = E^{\circ} - S \log a_{Na}$

Where:

E = measured cell potential in millivolts

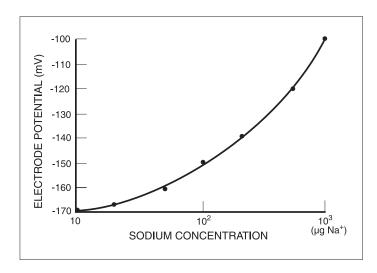
E° = "standard" electrode potential

S = 2.3RT/F, slope factor dependent on temperature

a_{Na} = activity of sodium ions

Typical electrode response to sodium ions from 10^{-1} M (2300 ppm) to 10^{-6} M (0.023 ppm) is shown in *Figure 6*.

Figure 6 Low Level Calibration Curve



5.2 Temperature Effects

The formula for potential given above indicates that the slope of the response, and therefore the actual potential, depends on the temperature. Normally all standards and samples should be measured at the same temperature (within 1 °C).

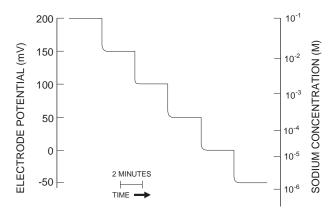
The change in the slope of the electrode response can be temperature corrected if manual calibration curves at different temperatures have been made. Some pH/mV/ion meters have a provision for entering the "isopotential" point (if known) for the electrode. This is the theoretical point at which all temperatures give the same potential. However, as the electrode standard potential and the slope change with temperature, correcting for just the slope is not accurate. The best method is to have a constant temperature for all measurements.

Very accurate work may require the use of a temperature bath for standards and samples. Maintain the temperature several degrees above the ambient temperature. With the sodium electrode, a 1°C change in temperature causes 0.3% change in slope.

5.3 Response Time

The response of the sodium electrode may take from 2–4 minutes to reach 99% of its total response for decade changes in sodium concentration. Response times with smaller concentration changes generally will be longer and can be minimized by proper stirring and conditioning. Experiment to determine the proper stir rate. Generally, conditioning the electrode in sodium standards (containing Sodium and Potassium ISA) near the concentration to be measured provides fastest equilibrium. *Figure 7* shows the electrode response for various changes in [Na+]. If the electrode response is slower than usual, try cleaning the glass membrane. See *Section 4.2 Cleaning the Electrode*.

Figure 7 Calibration Chart Recording



5.4 Ionic Strength Effects

The probe actually senses sodium ion activity, not concentration. The activity (the thermodynamic "force" of the ions) is related to the concentration:

$$a_{Na} = C_{Na} X f$$

Where:

a_{Na} = activity of sodium ions

C_{Na} = concentration of sodium ions

f = activity coefficient

The activity coefficient varies with ionic strength (a measure of total number of ions in the solution). Because the sodium concentration is usually what is measured, control of the ionic strength is a part of most procedures. Control the ionic strength by having all standards and samples at the same ionic strength. The activity coefficients are then equal for all measurements, and a calibration curve of concentration vs. potential (mV) can be plotted and used instead of activity vs. potential.

Raising the ionic strength to a high value by ionic strength adjustors is a common method of insuring that all samples have the same ionic strength. This high value minimizes the influence of sample ionic strength variation on sample measurement. Sodium ionic strength adjustors, which are concentrated salt solutions containing a base to raise the pH, are added to standards and samples before measurement.

The most commonly used ionic strength adjustor for sodium measurements is made from ammonium chloride and ammonium hydroxide. Hach offers an improved Sodium/Potassium ISA with a lower blank level, in convenient powder form, available in unit-dose powder pillows or bulk powder.

If a liquid ISA is preferred, the conventional ammonium ion based solution can be prepared as follows:

- **a.** Dissolve 20 g analytical reagent-grade ammonium chloride in 50 mL of deionized water.
- **b.** Add 27 mL of concentrated ammonium hydroxide.
- **c.** Dilute to 100 mL with deionized water. This ISA is used by adding 1 mL ISA per 50 mL of sample or standard.

5.5 Interferences

The sodium glass membrane responds to ions other than sodium, so interferences exist. Typically, electrode response to another ion increases the potential, causing a positive error. The response to other ions can be semi-quantitatively determined through the Nicholsky equation, an extended Nernst equation:

$$E = E^{\circ} + (RT/zF) \ln [a_{Na} + K_{NaX} X a_{X}]$$

Where:

 a_X = the activity of the interfering ion

 K_{NaX} = the selectivity coefficient for the interfering ion relative to sodium.

The value of the selectivity coefficient should be small to reduce the interference. A major advantage of the Hach Sodium electrode is the reduced interferences from other ions compared to the standard sodium selective glass (NAS 11–18). If the electrode is exposed to high levels of interferences, soaking in Sodium Electrode Storage Solution or 1 M sodium chloride will help remove the adsorbed ions from the glass membrane.

The major interferences are silver and hydrogen ions. Hydrogen ion concentration is reduced by the ISA, which raises the pH. If the samples are extremely acidic, or have a high buffer capacity, check that the sample pH is above 9 after adding ISA. If necessary, ammonium hydroxide can be added to the samples to raise the pH. The electrode potential will not be affected if the [H+] is about 1000-fold less than the [Na+], or the pH should be 3 to 4 units higher than the pNa.

Selectivity coefficients at 10⁻⁵ M Na⁺ (mixed solutions) are:

$$K_{Na^{+}/K^{+}} = 5 \times 10^{-5}$$
 $K_{Na^{+}/NH_{4}^{+}} = 2 \times 10^{-5}$
 $K_{Na^{+}/Aq^{+}} \ge 10$

SECTION 6 TROUBLESHOOTING

6.1 General Troubleshooting

The most important principle in troubleshooting is isolating the components of the system and checking each in turn. The components of the system are the 1) Meter, 2) Electrode, 3) Standard, 4) Sample, and 5) Technique. Also refer to *Table 3*, *Troubleshooting Checklist*.

Meter

The meter is the easiest component to eliminate as a possible cause of error. Hach meters have an instrument checkout procedure in the instruction manual and a shorting strap for convenience in troubleshooting. Consult the manual for complete instructions and verify that the instrument operates as indicated and is stable in all steps.

Electrode

- 1. Rinse the electrode thoroughly with demineralized water.
- **2.** Check the electrode operation (slope). If the electrode fails this procedure, re-soak the sodium electrode as directed in *Section Section 1.3.2 Preparing the Reference Half Cell*.
- **3.** Repeat the slope check in *step 2*.
- **4.** If the stability and slope check out properly, but measurement problems persist, the sample may contain interferences or complexing agents, or the technique may be in error. See the *Standard, Sample, and Technique* sections, below.
- **5.** Before replacing a "faulty" electrode, or if another electrode is not available for test purposes, review the instruction manual and be sure to:
 - Clean the electrode thoroughly.
 - Prepare the electrode properly.
 - Use proper filling solutions, ISA, and standards.
 - Measure correctly.
 - Review the Troubleshooting Checklist.

Standard

The quality of results depends greatly upon the quality of the standards. ALWAYS prepare fresh standards when problems arise—it could save hours of frustrating troubleshooting! Errors may result from contamination of prepared standards, quality of dilution, deionized water, or a numerical error in calculating the concentrations. Do not confuse sodium and sodium chloride standards.

The best method for preparing standards is serial dilution. An initial standard is diluted, using volumetric glassware, to prepare a second standard solution. The second is similarly diluted to prepare a third standard, and so on, until the desired range of standards has been prepared. Hach has a variety of sodium standards available.

Sample

If the electrodes work properly in standards but not in the sample, look for possible interferences, complexing agents, or substances that could affect response or physically damage the sensing electrode or the reference electrode. If possible, determine the composition of the samples and check for problems. See *Section Section 5.5 Interferences*.

Technique

Check the method of analysis for compatibility with your sample. Direct measurement may not always be the method of choice. If a large amount of complexing agents are present, known addition may be best. If the sample is viscous, analate addition may solve the problem.

If working at low levels, follow the low-level measurement technique. Also, be sure that the expected sodium concentration is within the electrode's limits of detection.

If problems persist, review operational procedures and instruction manuals to be sure that proper technique has been followed. Reread *Section 1.6 Measuring Hints* and the procedures section.

Table 3 Troubleshooting Checklist

Symptom	Possible Cause	Remedy
Slow Response	Glass needs to be conditioned	Soak in storage solution or standard with ISA.
	Glass bulb is coated	Clean bulb.
	Corroded glass bulb	Soak in hydrochloric acid for 15 minutes. Ensure the reference electrolyte tube is capped on combination electrodes. Recondition the electrode as described in Cleaning the Electrode.
	A pH electrode is connected to the 5-pin connector.	Disconnect pH electrode.
Low slope or no slope	Standards made incorrectly	Prepare new standards and check procedure.
	Glass bulb or internal stem is cracked	Check electrode response; replace electrode if necessary.
	pH is too low	Verify pH is > 9 after each ISA addition.
	ISA not used	Add ISA to each Sample and standard.
	Insufficient conditioning	Condition overnight in storage solution.
Noisy, unstable readings	Samples and standards at different temperature	Allow solutions to come to room temperature slowly before measuring.
	Electrode not connected to meter	Ensure electrode is properly connected.
	Air bubbles in reference electrolyte tubing	Purge air bubbles-depress and turn dispenser knob several times.
	A pH electrode is connected to the 5-pin connector.	Disconnect pH electrode.
Drift in reading	Temperature of sample is changing	Allow samples and standards to reach the same temperature.
	Incorrect reference electrolyte used	Ensure Ammonium Chloride Electrolyte gel is being used.
	Glass bulb is coated	Clean electrode.
	A pH electrode is connected to the 5-pin connector.	Disconnect pH electrode.

ELECTRODE SERVICE REQUEST QUESTIONNAIRE

- 1. What is the catalog number of the electrode?
- **2.** What model of meter is the electrode being used with?
- **3.** What is the complete lot code of the electrode (located on the electrode cable)?
- **4.** What is the date the electrode was purchased?
- **5.** How long has the electrode been in use?
- **6.** What types of samples are being tested?
- **7.** What is the temperature of the samples being tested?
- **8.** How often is electrode being used?
- **9.** How is the electrode being stored between uses?
- **10.** What are the millivolt readings in standards that are a decade apart for ISE electrodes?
- **11.** What is the slope of electrode during a typical calibration?
- **12.** If the electrode is new, has it been conditioned according to the manual?
- **13.** If the electrode has been in use, what maintenance has been performed as suggested in the manual?
- **14.** Describe the suspected problem or failure of the electrode.
- **15.** When calling for telephone support it will be helpful to have your meter, electrode, and buffers/standards, along with this completed questionnaire, near the phone before calling.
- **16.** Did you use ISA in all samples and standards?
- **17.** Did you use the low range method for drinking water samples below 3 mg/L sodium?
- **18.** Is the sample above 1.0 mg/L sodium (the linear range of the electrode)?

Acid — a chemical compound that contributes hydrogen ions (H+) to an aqueous solution or causes the pH to be less than 7.0.

Acidic — A solution or system with a pH less than 7.0

Activity — The measure of work performed by an ion as it moves through a system. The activity is dependent upon the concentration of all ions in the system and the charge associated with the ion.

Alkali — A solution or system with a pH greater than 7.0

Alkalinity — Capacity of water to accept hydrogen ions (H⁺). Indicates presence of carbonate (CO₃²⁻), bicarbonate (HCO₃), and hydroxyl (OH⁻) ions.

Anion — A negatively charged ion (i.e., NO₃-, Cl-).

Base — A chemical compound that contributes hydroxide ions (OH-) to an aqueous solution or causes the pH to be greater than 7.0

Buffer —

- 1) A compound, mixture of compounds, or solution which, when added to a system, changes pH to a specified, known value.
- 2) The ability of a solution or system to resist change in pH if either an acid or a base is added.

Buffering capacity — A measurement of the amount of acid or base which can be added to a solution before the pH changes.

Cation — A positively charged ion (i.e., NH₄⁺, Ca₂⁺).

Conditioning — The process of preparing an electrode for use by soaking the electrode in a solution with a pH and ionic strength similar to the sample. Conditioning allows the hydrated layer, necessary for accurate measurements, to form.

Decade — Any two standards with a concentration ratio of 10. For example:

Concentration of the higher standard Concentration of the lower standard = 10

Dilution factor — A numerical value by which results must be multiplied in order to obtain the actual value of the sample. For example, if 1 mL of sample is diluted to 10 mL, then the result obtained must be multiplied by 10 to obtain the value for the sample before dilution.

Electrolyte — A solution made of dissolved ions that is used to facilitate electrical conductance and ionic transport. The composition is usually potassium chloride (KCl), sodium chloride (NaCl), ammonium chloride (NH₄Cl), or ammonium sulfate $((NH_4)_2SO_4)$.

Free-flowing reference junction — The point on the electrode where reference gel is freely dispensed without obstruction by frits or other materials.

Hydrated layer — A layer that forms at the glass/liquid interface. Chemically similar to the sample being measured (especially in samples of low ionic strength), the formation characteristics of this layer depend upon the type of glass, the age of the glass, the previous usage history of the glass, the temperature and ionic strength of the aqueous solution, etc.

Inert — Does not react with other substances.

Ion — An atom, group of atoms or molecule that is electrically charged as the result of gaining or losing electrons. An ion resulting from the gain of electrons becomes negative (anion), while an ion resulting from the loss of electrons becomes positive (cation).

Ionic strength — A measure of the concentration of any ion in solution, usually expressed in moles per liter.

Occasional use — Infrequent use where the probe is subjected to test conditions just long enough to obtain a proper reading.

pH — A relative numerical measurement of the acidic, neutral, or alkali nature of a solution or system. Mathematically defined as the negative log of the hydrogen ion concentration.

GLOSSARY, continued

Potential — The capacity of a system to perform work. When two ions of opposite charge are separated by a distance, there is a potential developed from the tendency of the ions to come back together. Each ion exerts work to come back together. The amount of work needed by each ion to come back together is the potential.

Reference junction — The point on the electrode where reference gel is dispensed.

Routine use — Typical use following regular, frequent routines, but not continuous as with an in-line monitoring process.

Salt — An ionic compound.



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Catalog number

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Quantity

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