

SODIUM ION-SELECTIVE ELECTRODE

Updated September 2013

I. Reference:

AOAC 976.25

Adapted in 1990

Applicable to foods containing 100 mg Na/100 g or less

II. Personal Protective Equipment:

1. Lab coat
2. Safety glasses/goggles
3. Latex gloves

III. Reagents:

1. Sodium Standard Solutions:

a. Stock Solution: 10 mg Na/mL (2.5421 g NaCl dried overnight at 100°C into 100 mL ddH₂O)

b. Working Solution:

- i. **0.1 mg Na/mL:** Pipette 1 mL stock solution into 100 mL volumetric flask and dilute to volume with buffer solution (mix well).
- ii. **1 mg Na/mL:** Pipette 10 mL stock solution into 100 mL volumetric flask and dilute to volume with buffer solution (mix well).
- iii. **2 mg Na/mL:** Pipette 20 mL stock solution into 100 mL volumetric flask and dilute to volume with buffer solution (mix well).

Note: The concentration of the working solution to use depends on the expected concentrations of sodium in the samples.

mg Na/100g	% Na	Working Solution
0 to 5 mg	.00 to .005	.1 mg Na/mL
5 to 50 mg	.005 to .050	1 mg Na/mL
50 to 100 mg	.050 to .100	2 mg Na/mL

2. Buffer Solution A (1990 revision):

a. Dissolve 74.6 g Triethanolamine in approximately 700 mL ddH₂O and add 200 mL Sodium Ionic Strength Adjustor (Orion #841111).

b. Adjust pH to 9-10 with Sodium Ionic Strength Adjustor if necessary and dilute to volume (1 L) with ddH₂O.

3. Buffer Solution B (AOAC 976.25):

a. Dissolve 74.6 g Triethanolamine in approximately 900 mL ddH₂O and adjust pH to 10.2 (Accumet sheet suggests a pH of 9.5) with concentrated HCl.

b. Dilute to volume (1 L) with ddH₂O.

Note: Only one Buffer Solution (A or B) is needed

Note: If using liquid Triethanolamine, use 66.6 mL (d = 1.12 kg/L) of Triethanolamine.

IV. Electrode Preparation:

1. Rinse the electrode with dH₂O to remove crystal residue (caused by natural leakage during storage).
2. Slide the rubber sleeve down to expose the electrolyte filling hole.
3. Check electrolyte level in the reference cavity (outer space). Electrolyte level should be ~1/4 in. below the cap. If the electrolyte level is too low, add electrolyte (saturated KCl).
4. Proper electrode function requires electrolyte flow at the junction. Always check for proper electrolyte flow at the junction before using electrode to test samples (see Accumet instruction sheet for more details).
 - a. Hold electrode upright at a 45° angle between the thumb and forefinger of the left hand with the fill hole facing away from you.
 - b. Insert spout of the KCl bottle into the fill hole and create an airtight seal (apply firm pressure).
 - c. Squeeze bottle to check the silicon bung at the base of the electrode (white dot or rod).

V. Check Electrode Function:

1. Pipette 1 mL of sodium standard (either .1 M or 1000 ppm) into beaker.
2. Add 99 mL of buffer.
3. Stir solution approximately 30 seconds and record the millivoltage.
4. Pipette 10 mL of sodium standard (either .1 M or 1000 ppm) into same beaker.
5. Stir solution approximately 30 seconds and record the millivoltage.
6. Correct electrode function is indicated by a difference of 59 +/- 4 mV.
7. Electrode will need to be rejuvenated (see Accumet instruction sheet) if the difference falls out of this range.

VI. Blank Determination/Standard Curve:

1. Pour 100 mL of buffer solution into a 150 mL beaker and stir for 3 minutes with electrode immersed in solution.
2. Record millivoltage.
3. Add 1 mL of working solution to beaker, stir for 30 s, and record mV.
4. Continue to add 1 mL portions of working solution, stir for 30 s, and record mV to establish the blank (0 reading) and standard curve.

VII. Sample Preparation:

1. Weigh 5.0 g of sample (1-mm grind) into 150 mL screw cap flask
2. Pour 100 mL of buffer solution into sample jar and stir magnetically 3 minutes with electrode immersed in solution to equilibrate.
3. Record mV potential.

VIII. Calculations:

1. Plot mg Na (x axis) and mV potential (y axis) of standards omitting the 0 (blank) reading to establish a standard curve.
2. Using the equation of the line, calculate the mg Na in the sample and the mg of Na in the blank using the corresponding mV readings.

Note: The standard curve may be either logarithmic or linear.

$$\text{Mg Na/100 g feed} = \frac{(S - B) * 100}{W}$$

S = mg Na in sample

B = mg Na in blank

W = g of sample