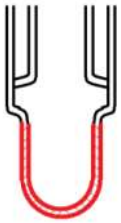
Sodium Glass Combination ISE Manual



WORKING PRINCIPLE:

The Sodium glass ion-selective electrodes work similar to pH glass electrodes: the chemistry of the glass sensing membrane is altered to have sodium ions (Na+) selectivity rather than for hydrogen ions (H+), as in the case of pH electrodes.

However, both the pH electrodes and the glass Sodium ISE have a similar set-up and working principles. In aqueous solutions, cations are removed from the outer hydrated layer of the sensing glass and replaced by sodium ions (Na+) from the sample. This creates a potential at the interface between the sample solution and the membrane, which in turn depends on the activity of the sodium. The internal solution contains a fixed concentration of the cation of interest and therefore fixes the internal electrode surface potential, while that in the external solution varies.

The electrochemical potential develops only in each hydrated gel layer based on an ion-exchange principle that leads to a phase boundary potential.

Specification Sheet:

Slope at 25˚C:	+54/+66 mV (100 – 1000ppmstandards)	**Main Interferences (for 10^{-3}M Na^+):**	$3x10^{-8}$ Ag^+, $5x10^{-3}$ Li^+, 0.1 K^+ , 0.5 Ti^+
Reproducibility:	+/- 2%	**Required Reference:**	Double Junction
Response Time:	95% response in 30seconds	**Body Materials:**	Glass
Potential Drift:	2mV Per Day	**Reference Electrolyte:**	0.1M NH_4Cl Solution
Concentration Range (mol/l):	10^{-7} - 3	**ISAB:**	SISAB Solution
Limits (ppm):	0.002-69,000	**Dimensions:**	12x120mm
Temperature Range (˚C):	5-80˚C	**Plug Type:**	BNC Other Requests Available
Pressure Range (Bar):	Ambient Only	**Storage:**	NaCl Solution
pH Range:	9-12		

Required Test Solutions

SOLUTION LIST FOR SAMPLES WITH IONIC STRENGTH >10^{-2} M*:

1. Standard Stock Solution: 1000ppm standard: 2.542g NaCl in 1L of solution prepared with DI water

2. Fill Solution: 0.1M Ammonium Chloride Solution

3. ISAB: Place 20g reagent grade Ammonium Chloride in a 100ml volumetric flask. Dissolve in aorund 50ml DI water. Add 27ml concentrated Ammonium Hydroxide and dilute to volume with DI water. Check the pH of the solution is as close to 12 as possible – if it is below this, add more Ammonium Hydroxide

4. Rinsing Solution: Diluted ISAB (add 20ml ISAB to 100ml flask, adjust to 100ml with DI water)

5. Storage Solution: 0.01 Molar Sodium Chloride solution

SOLUTION LIST FOR LOW IONIC STRNGTH SAMPLES (<10^{-2} M)*:

1. Standard stock solution: prepare appropriate level standards corresponding to sample concentration, by serial dilution. Add 1ml low level ISAB (see below) to 100ml of standard

2. Fill solution: Dilute 20ml of outer chamber fill solution (0.1M Ammonium Chloride Solution) to 100ml with DI water

3. ISAB: Follow the standard ISAB instructions, then dilute 20ml ISAB to total volume 100ml using DI water.

4. Rinsing solution: diluted low level ISAB (add 20ml low level ISAB to 100ml flask, adjust to 100ml with DI water)

5. Storage solution: 0.01 Molar Sodium Chloride solution

***All solutions should be prepared using high purity/lab grade chemicals and DI water**

Electrode Visual Check:

SILVER WIRES:

- make sure the wires are not tarnished/black – this indicates contamination;

- the AgCl dip should be metallic and shiny. If grey and matt, this is a sign that the wire is deplating.

ELECTROLYTES:

- check that the electrode is not empty and the electrolyte is clear. If not, use the fill hole to empty, clean and refill with new electrolyte.

JUNCTION:

- check that the junction is not locked. If it is, try to clean with appropriate solvent, rinse thoroughly with DI water and rehydrate overnight.

GLASS MEMBRANE:

- make sure the membrane is not cracked or scratched;

- check if the membrane is clean and clear. If it is coated, clean with appropriate solvents, rinse with DI water and rehydrate.

ELECTRODE SLOPE CHECK:

The SLOPE is defined as the change in potential observed when the concentration changes by a factor of 10.

- In a beaker, add 100mL DI water + 2mL ISAB

- Add 1mL 1000ppm standard, homogenize the solution, and take a reading = R1 (it can take up to a few minutes)

- Add 10mL 1000ppm standard, homogenize the solution, and take a reading = R2

- Determine the difference between the two readings (R1-R2) – an interval of 54 - 66 mV indicates correct electrode operation, if the measuring temperature is 20-25˚C

- When testing, take in account the measuring temperature, as values differ markedly, as seen in the table.

Variation Of Slope With Temeprature

Temperature (˚C)	Slope (mV)
0	54.20
10	56.18
20	58.16
25	59.16
30	60.15
40	62.13
50	64.11

OPERATIONAL TIPS:

- Ensure the pH of the standards and sample are between 9-12. If this is not the case, adjust the pH using ammonium hydroxide;
- Allow the standards, sample and electrode to equilibrate at the same temperature prior to test – a difference of 1˚C will result in approx. 2% error. Ensure stirrer does not heat and change the temperature of the solutions (due to friction or electrostatics) – isolate beaker from stirrer by putting a piece of carboard or styrofoam in-between;
- Prepare fresh solutions daily, ensuring all equipment is clean and gloves are worn to avoid contamination. Plastic labware is recommended;
- Choose standard concentrations one decade away (eg. 10ppm and 100ppm) that bracket your expected sample concentration;
- Typical addition would be 1ml of ISAB to 100ml of standard solution, however the ionic strength of the standard and solution should be kept constant between all standards and samples;
Start with lowest concentration of standard to avoid cross contamination;
- The ideal measurement is static after mixing/stirring. In many cases it is not possible but variable stirring rates means changes in activity which should be avoided as sensors measure activity. Once the reading has stabilised, record the value;
- Repeat steps above for the second standard;
- New calibration curves should be performed daily;
- Ensure the electrode is rinsed with the correct rinsing solution between measurements, and tap dry (do not rub glass membrane);
- Store in storage solution;
- Do not store dry or in DI water as this will dehydrate the hydrated glass layer;
- Do not touch or rub the glass membrane to avoid coating the membrane.

INTEFERENCES:

If certain cations are present in high enough concentrations, they will cause errors in measurement. Using ammonium ion in the recommended ISAB does not result in an error provided all standards and samples have the same level of ISAB added. In cases where interferences are high, the electrode may become drifty and sluggish. If this occurs, soak the electrode in storage solution.

Table to show levels of possible interferences causing 10% error at various levels of sodium:

	Molar			Parts Per Million		
Ion	**10^{-4} $Na+$**	**10^{-3} Na^+**	**10^{-2} Na^+**	**1ppm Na^+**	**10ppm Na^+**	**100ppm Na^+**
Li^+	$5x10^{-4}$	$5x10^{-3}$	$5x10^{-2}$	2	15	150
K^+	$1x10^{-2}$	0.1	1	170	1700	17000
Rb^+	0.3	3	-	11000	110000	-
NH_4^+	0.3	3	-	1800	18000	-
Ag^+	$3x10^{-9}$	$3x10^{-8}$	$3x10^{-7}$	10^{-4}	10^{-3}	10^{-2}
Tl^+	$5x10^{-2}$	0.5	-	4500	45000	-

Measuring Techniques

DIRECT POTENTIOMETRY:

A. CALIBRATION

1. Prepare a series (at least 2) of standards that bracket the expected sample concentration using serial dilution. Ideally standards should be a decade in concentration apart e.g. 1, 10, and 100ppm

2. Dispense 50 ml of each standard into analytically clean beakers

3. Add ISAB/TISAB in the appropriate ratio. As a guide with sample concentrations in the 1 to 1000ppm range, 1ml of ISAB to 50 ml sample is satisfactory

4. Rinse the electrode with DI water and blot dry with a lint free cloth and place in the lowest standard. Take the calibration reading.

5. Repeat steps above for all subsequent standards proceeding from lowest to highest

B. SAMPLE MEASUREMENT

6. Rinse the electrode in DI water and blot dry. Place the electrode in the sample

7. Record the sample reading

C. CLEANING AND STORAGE

8. Rinse the electrode in appropriate solvent (that dissolves the sample), rinse in DI water and blot dry.

9. Place the electrode in storage solution.

KNOWN ADDITION – for samples in which the matrix is not entirely clean or aqueous, hence comparison with clean standards is not appropriate.

- Measure the potential of the sample solution
- Add a small volume of a higher concentration standard solution and measure the potential [Typical sample volume is 50 ml, typical standard volume is 5 ml. The standard should be approximately 100 time the sample concentration for accurate analysis]
- From difference in the two values, and using the known electrode slope, the unknown concentration is determined.

SAMPLE ADDITION – ideal for dirty or viscous samples with a complex matrix. The sample however needs to be relatively concentrated i.e. at least 100 times the electrodes linear detection limit. The analysis does have the benefit of only requiring a small volume.

- Measure the potential of a dilute standard solution
- Add a small volume of more concentrated sample and record the potential
- Note the difference between the measurement and using this value (and the predetermined electrode slope) the unknown concentration is determined
- The sample matrix is basically broken down by dilution with the standard and therefore analysis is carried out in the same media.

*Make sure all solutions are set up at the same temperature (or within ±2˚C);
**Mix/stir well the solutions, but perform a static measurement.

Troubleshooting

Symptom	Possible Causes	Next Step
Off Scale or over range reading	Defective Meter	Check meter is not shorting (see meter instructions)
	Electrode not plugged in properly	Unplug electrodes and reset meter
	Junction is dry	Soak the electrode in warm storage solution
	Electrode is not filled	Refill electrode with appropriate filling solution
	pH too low	Adjust pH level
	Oils/fats deposit on the sensing membrane	Soak in a solution with a mild detergent, rinse with DI and recondition
	Incorrect standard used	Prepare fresh / change standards
	Insufficient conditioning	Condition for minimum 8 hours storage solution
Noisy or unstable readings	Defective meter	Check meter is not shorting
	ISAB not used	Use recommended ISAB
	Stirrer not grounded	Ground stirrer
	Electrode is exposed to interferences	Soak overnight in storage solution
Drift (Slow change in one direction)	Samples and standards at different temperatures	Allow solutions to equilibrate to room temperature before measurement
	Incorrect filling solution	Empty fill solution, rinse and refill with correct solution
	Contaminated glass bulb	Clean membrane and recondition
	Clogged reference junction	Clean the junction, rinse with DI and recondition
	Membrane dehydrated	Soak overnight in storage solution
	pH too low	Adjust pH as close to pH12 as possible, ensuring the chemical used to do this does not interfere with the electrode
	ISAB not used	Use recommended ISAB to stabilise the reading

Troubleshooting Continued

Symptom	Possible Causes	Next Step
Low slope or no slope	Standards contaminated or incorrectly made	Remake fresh standards, avoid any sources of sodium contamination
	ISAB not used	Use recommended ISAB
	pH too low	Adjust pH as close to pH12 as possible, ensuring the chemical used to do this does not interfere with the electrode
	Membrane dehydrated	Soak overnight in storage solution
	Air bubbles on the membrane	Shake electrode as a thermometer, mix well in the beaker
Reading Same in all solutions	Electrical issue	Contact Tech Support
Calibration curve is ok but reading seems incorrect	Incorrect standards	Ensure the correct standards are used, double check dilutions and ensure the standards are appropriate for the level of the sample
	Incorrect units used	Apply correct conversion factor: 10^{-3} M =23ppm
	Sample pH too low	Adjust pH as close to pH12 as possible, ensuring the chemical used to do this does not interfere with the electrode
	Possible interferences	Look for any interference sources, complexing agents or substances that can affect the response or physically damage the sensor
Slow stabilisation times	Contaminated electrode	Clean using appropriate solvent, rinse and rehydrate
	Contaminated fill solution	Clean and refill
	Low sample temperatures or temperature differ between samples	The lower the temperature and the greater the difference between samples, the longer the response time – ensure same temperature
	Air bubbles in the reference cell	Gently shake the electrode like a thermometer