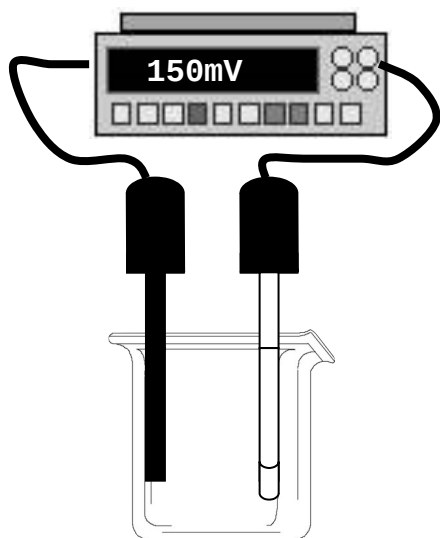
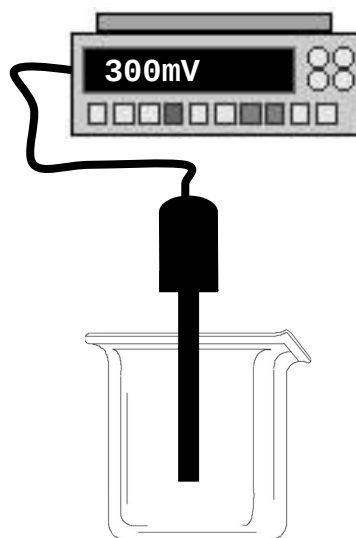


SENTEK

Ion Selective Electrode OPERATING INSTRUCTIONS



Mono/Reference



Combination

SENTEK

Unit 6 & 7 Crittall Court, Crittall Drive, Springwood Industrial
Estate, Braintree, Essex, CM7 2SE

Tel: +44 (0) 1376 340 456 Fax: +44 (0) 1376 340 453 Email: sentekuk@btconnect.com

S
O
D
I
U
M

Operating Instructions

Sodium Ion Selective Electrode

This Document includes operating instructions for two types of Ion Selective Electrode. A single sensing Half Cell Sodium Electrode which requires a separate Reference Electrode, and a Combination Sodium Electrode which has the Reference built into the outer shaft.

For extremely low level work the Half Cell plus Reference Electrode should be used. For general applications the Combination is more convenient.

Depending on the type of ISE chosen you will need to disregard some information for instance if you have the Combination Electrode then disregard any comments about reference electrodes.

A quick guide is included with a more involved section following.

SENTEK

Unit 6 & 7 Crittall Court, Crittall Drive, Springwood Industrial

Estate, Braintree, Essex, CM7 2SE

Tel: + 44 (0) 1376 340 456 Fax: +44 (0) 1376 340 453 Email: sentekuk@htconnect.com

Quick Guide to Sodium ISE

The combination ion selective electrode has a solid state PVC polymer membrane with an integral driTEK reference electrode. The electrode is designed for the detection and analysis of Sodium ions in aqueous solutions and is suitable for use in the field in the laboratory and in on line analyzers.

Installation

Connect the ISE to the mV or ion meter.

Remove the black protective cap and keep it in a safe place.

The ISE can be used immediately but pre soaking for 5 minutes in a 100ppm Sodium solution is recommended.

The ionic strength of the standards and solutions should be kept constant between all standards and samples. This is achieved by the simple addition of an Ionic strength adjustment buffer (ISAB). Lithium Acetate is ideal. A typical addition would be 1 ml of 1 molar ISAB to 100 ml of standard and sample.

For low level measurements below around 50ppm in relatively pure samples no ISAB is needed.

No temperature correction is necessary however standards and samples should be measured at the same temperature.

Begin calibration from the lowest concentration standard to avoid cross contamination. Calibration should cover the anticipated range of the samples.

Rinse tip with de-ionised water between measurements.

Avoid strongly acidic or alkaline samples, strong detergents and organic solvents.

Storage and Maintenance

Avoid touching the membrane surface.

After use rinse with de-ionised water, replace protective cap and store dry in its box.

If performance becomes sluggish, rinse with dilute detergent, rinse with de-ionised water and immerse the tip in a 1000ppm Sodium solution for 1 hour.

Specification

Sodium is a monovalent cation. A 1 molar solution of Sodium contains 23.00 grams per litre of Sodium.

A 1000ppm Sodium solution is equivalent to 0.043 moles per litre

Refer to the table below for the full specification

Parameter	Specification
Overall length	155 mm
Body Diameter	12 mm
Cap Diameter	16mm
Connector	BNC
Cable length	1000 mm
Resistance at 25 Deg C	< 200 Meg Ohm
Concentration Range	0.05 to 20,000 ppm
Slope	54 to 59 mV per decade
Potential Drift	2 mV per day
Operating pH range	
Temperature range	5 to 50 Deg C
Endpoint time	Typically 10 to 30 seconds
Interferences: Ions with coefficients above 0.001.	Potassium Ammonium Calcium Magnesium

Recommendations for successful analysis

The combination ISE's can be used with any pH/mV meter or Ion meter. If the meter does not have a BNC socket and you have a BNC electrode please contact your distributor who will arrange to have the correct plug fitted. Adapters are also available if the same electrode has to be used on more than one meter.

Meters with a 0.1 mV resolution are recommended whilst dedicated Ion meters will provide direct concentration readouts saving time and effort in constructing calibration curves and performing calculations. Your distributor can advise on the most suitable meter.

Magnetic stirrer/stirrer bars are recommended for laboratory analysis. Please operate at the lowest constant speed available.

Semi-logarithmic (4-cycle) graph paper is required for preparing calibration curves when you are using a mV meter.

Required Solutions

Distilled or de-ionised water will be required to prepare Standards, ISABs and to rinse the electrode between measurements.

1000ppm Stock Standard solution. Used for preparation of Standards.
(Prepared by customer)

ISAB. Used to adjust the Ionic strength of all standards and samples. Typical addition is 1 ml of 4 Molar ISAB to 50ml of all standards and samples.

Operation

1. Connect the electrode to the meter being used for analysis
2. Prepare a series (at least 2) of standards that bracket the expected sample concentration. This is best done by serial dilution of the stock solution. Ideally standards should be a decade in concentration apart e.g. 1, 10, and 100ppm.
3. Dispense 50 ml of each standard into analytically clean beakers (100 to 150 ml size is perfect)
4. Add ISAB/TISAB in the appropriate ratio. As a guide with sample concentrations in the 1 to 1000ppm range 1ml of a 4 Molar ISAB to 50 ml sample is satisfactory. For TISAB(Fluoride analysis only) please read the label.
5. Rinse the electrode with de-ionised water and blot dry with a lint free cloth and place in the lowest standard. When the reading is stable record the mV value.
6. Repeat step 6 for all subsequent standards proceeding from lowest to highest.
7. Plot a calibration curve on semi log paper using mV values on the linear Axis and concentration on the log scale.
8. Rinse the electrode in de-ionised water and blot dry. Place the electrode in the sample and record the stable mV value.
9. Using the calibration curve determine the unknown sample concentration.

Hints and tips

1. Ensure that the temperature of all standards and samples are the same to reduce errors.

2. Using a magnetic stirrer for laboratory analysis is recommended but not essential. It is however important to have the stirrer set on a low constant speed which must be reproducible for all measurements.
3. Prior to sample measurement ensure that the electrode is thoroughly rinsed with de-ionised water. It is worth performing this rinse twice given the possibility of carryover being greatest in high concentration solutions.
4. Prepare standards by serial dilution.
5. Make sure your electrode is conditioned by leaving the tip in the lowest concentration standard for 1 hour prior to analysis.

Methods of Analysis

We have described direct potentiometry above. This method is simplified by using a direct reading ion meter. There are several other methods, which are useful.

Known Addition: An incremental technique where the potential of the sample solution is measured followed by addition of a small volume of a higher concentration standard solution. The new potential is measured and from difference in the two values, and using the known electrode slope, the unknown concentration is determined,

This method is ideal for samples whose matrix is not entirely clean or aqueous. In these instances comparisons with clean standards is not appropriate thereby making direct potentiometry unsuitable. Known addition works because both standard and sample are measured in the same matrix.

Typical sample volume is 50 ml, typical standard volume is 5 ml. The standard should be approximately 100 times the sample concentration for accurate analysis.

Sample Addition: An incremental technique where the potential of a dilute standard solution is measured followed by the addition of a small volume of more concentrated sample. The new potential is recorded and the difference noted. Using this value (and the predetermined electrode slope) the unknown concentration is determined.

This method is ideal for dirty or viscous samples with an awkward 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.

The sample matrix is basically broken down by dilution with the standard and therefore analysis is carried out in the same media.

End Point Titration: Flow Plus combination ISE's are ideal end point indicators and will produce a significant potential change at the equivalence point. The Ion in question must be contained in the titrand or the titrant and must therefore be in excess or absence at the end point.

Operating Instructions for Sodium ISE

Contents

General Information

Introduction
Required Equipment
Required Solutions
Before using the Electrode
Connecting Electrode to Meter
Checking Electrode Slope
Digital pH/mV Meter

Using the Electrode

Units of Measurement
Sample Requirements
Analytical Procedures
Direct Measurement Using Digital pH/mV Meter
Low-Level Measurement Using Digital pH/mV Meter
Known Addition Using Digital pH/mV Meter
Measuring Hints

Trouble Shooting Checklist

Electrode Characteristics

Electrode Response
Reproducibility
Interference's
Limit of Detection
Temperature Effects
pH effects
Electrode Storage
Theory of Operation

Specifications

The 315-75 Single Sensing Sodium electrode allows sodium in aqueous solutions to be measured quickly, simply, accurately and economically. All apparatus and solutions required for measurements, general analytical procedures, electrode characteristics, and electrode theory are discussed in this manual.

The 315-75C houses both a sodium sensing electrode and a calomel reference electrode in one body. The sodium sensing portion of the electrode is identical to the 315-75.

Required Equipment

Meter - Digital pH/mV meter or specific ion meter for laboratory, field or plant measurements.

Reference Electrode - (For use with 315-75 only) - Double Junction Ag/AgCl reference electrode, part no. R2. Do not use outer chamber filling solution provided with the double junction reference electrode.

Magnetic Stirrer - Recommended for laboratory measurements.

Graph Paper - Semi-logarithmic paper to prepare calibration curves.

Required Solutions

Distilled or De-ionised Water - To prepare all solutions and standards.

Standard Solutions - 0.1M Sodium chloride standard solution.

1000ppm standard (for measurement in units of ppm) - To prepare, transfer 43.5ml 0.1M standard into a 100ml volumetric flask and dilute to volume with distilled water.

Ionic Strength Adjuster: (ISA)

To keep a constant background ionic strength and adjust the pH. To prepare the ISA, place 20g reagent-grade ammonium chloride in a 100ml volumetric flask. Dissolved in about 50ml distilled water, add 27ml concentrated ammonium hydroxide, and dilute to volume with distilled water.

Reference Electrode Filling Solutions.

Inner Chamber - SAT KCl

Outer Chamber - 0.1M NH_4Cl . To prepare, dissolve 0.5g reagent-grade NH_4Cl in 100ml distilled water.

Combination Electrode Filling Solution
0.1 M NH_4Cl

Electrode Storage Solution 1ppm Sodium Solution.

Electrode Rinse Solution Dilute ionic strength adjustor (ISA). To prepare, add 20ml ISA to one litre volumetric flask and dilute to volume with distilled water. Use to rinse electrode between measurements.
DO NOT USE DISTILLED WATER.

Before using the Electrode Remove protective cap covering the tip of the electrode. Fill reference or combination electrode with appropriate filling solution.
SOAK OVERNIGHT IN ELECTRODE STORAGE SOLUTION.

Connecting Electrode to Meter
Insert the reference electrode phone-tip connector and the sensing electrode connector into appropriate jacks on the digital pH/mV or

specific ion meter. Consult your meter instruction manual.

Checking Electrode Slope with Digital pH/mV Meter.

Note: Check Electrode daily.

1. Put 100ml distilled water and 2ml ISA into a 150ml beaker. Turn function switch to mV position. Place electrodes in the solution after rinsing with electrode rinse solution. If drift or unstable readings are observed, see the troubleshooting checklist.
2. Pipette 1ml 0.1M or 1000ppm standard into the solution. Stir thoroughly. When reading is stable, read electrode potential in millivolts and record.
3. Add 10ml 0.1M or 1000ppm standard. Stir thoroughly. When reading is stable, read electrode potential in millivolts and record. Determine difference between the first and second potential readings. Correct electrode operation is indicated by a difference of 56 ± 2 mV assuming the solution temperature is between 20°C and 25°C. If the change in potential is not within this range, see the troubleshooting checklist.

NOTE: The above procedure measures electrode slope. Slope is defined as the change in potential observed when the concentration changes by a factor of ten.

Using the Electrode

Units of Measurement.

Sodium can be measured in units of moles per litre, parts per million, or other convenient units, depending on the concentration units used for standard solutions. (See table 1)

Samples Requirements

Samples must fall in the pH range of 9 to 12. For best accuracy, use the recommended ISA to adjust the pH. Samples and standards should be at the same temperature. The glass electrode body is resistant to attack by most organic solvents.

Analytical Procedures.

Direct measurement is a simple procedure for measuring a large number of samples. Only one meter reading is required for each sample. The temperature of samples and standards should be the same and the ionic strength of samples and standards should be made the same by addition of ISA to all solutions.

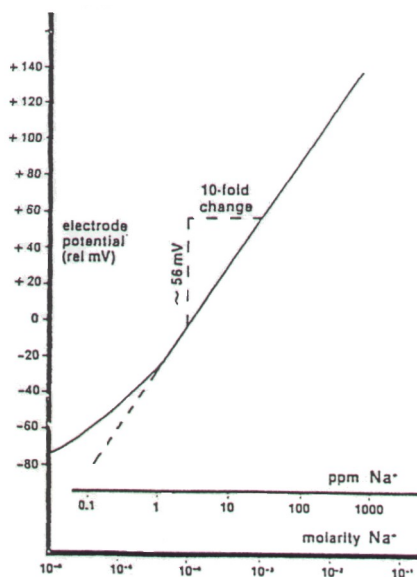
Direct measurement results can be verified by a known addition procedure. The known addition procedure involves adding a standard of known concentration to a sample solution. From the change in electrode potential before and after addition, the original sample concentration is determined.

Known addition is also a useful method for measuring occasional samples, as the preparation of a calibration curve is not required. As in direct measurement, any convenient concentration unit can be used.

Table 1
Concentration Unit Conversion Factors

<u>Moles per Litre</u>	<u>ppm as Sodium</u>
10^{-4}	2.30
10^{-3}	22.99
10^{-2}	229.9

Figure 1
Typical Sodium Electrode Calibration Curve



In the direct measurement procedure with a pH/mV meter a calibration curve is constructed on semi-logarithmic paper. Electrode potentials of standard solutions are measured and plotted on the linear axis against their concentrations on the log axis. In the linear regions of the curves, only three standards are needed to determine a calibration curve. In non-linear regions, more points must be taken. The direct measurement procedures in this manual are given for Low-level measurement procedures are given for measurements in the non-linear region.

Please note that the electrode potential corresponding to a particular sodium concentration on the curve is relative. It will vary from electrode to electrode.

Using Digital pH/mV Meter

1. For measurements in units of moles per litre prepare 10^{-2} , 10^{-3} , and 10^{-4} M standards by serial dilution of the 0.1 M standard solution. Add 2ml ISA per 100ml standard.
For measurements in units of ppm, prepare 100 and 10 ppm standards by serial dilution of the 1000ppm stock solution. Add 2ml ISA per 100ml standard.
2. Please electrodes in the 10^{-4} M or 10ppm standard after rinsing with electrode rinse solution. Set function switch to mV. Stir thoroughly, wait for a stable reading, and record.
3. Rinse electrodes with electrode rinse solution, and place in the 10^{-3} M or 100ppm standard. Stir thoroughly, wait for a stable reading, and record.
4. Rinse electrodes with electrode rinse solution, and place in the 10^{-2} M or 1000ppm standard. Stir thoroughly. Wait for a stable reading, and record.
5. Plot the millivolt readings (linear axis) against concentration (log axis) on standard semi-logarithmic paper. The curve may be extrapolated down to 5×10^{-5} M or 1ppm. Below this region, follow instructions for low-level measurements. See typical calibration curve in Figure 1.
6. Transfer 50 to 100ml sample to a 150ml beaker. Add 2ml ISA per 100ml sample.
7. Rinse electrodes with electrode rinse solution, and place in sample. Stir thoroughly. Record the millivolt reading when stable. Determine the unknown concentration from the calibration curve.
8. Re-standardise after 1-2 hours of use. Simply place electrodes in the 10^{-4} M or 10ppm standard and repeat steps 2-5 above.

Low-level Measurements

The following procedure is for solutions with a total ionic strength less than 10^{-2}M . For solutions low in sodium but high in ionic strength, perform the same procedure with one change; prepare a calibration solution with a composition similar to the sample.

Note: Use plastic laboratory ware.

Using Digital pH/mV Meter.

Note: Use plastic laboratory ware.

1. Prepare low-level ISA by diluting 20ml ISA to 100ml with distilled water.
2. If a double junction reference electrode is used, dilute 20ml outer chamber filling solution to 100ml with distilled water and fill the reference electrode.
3. Prepare calibration solution. For measurements in ppm, add 1ml low-level ISA to 100ml of the 100ppm standard. For measurements in moles per litre, prepare 100ml 10^{-3}M standard by hundred-fold dilution of the 0.1M solution. Add 1ml low-level ISA. Prepare standards fresh each day.
4. Place 100ml distilled water in 150ml beaker. Add 1ml low-level ISA. Adjust pH above 9 with NH_4OH , if necessary. Place electrodes in solution after rinsing with electrode rinse solution. Stir thoroughly.

Set function switch to mV. Add increments of the 100ppm or 10^{-3} M standard to the ISA solution using steps outlined in [Table 2](#). Record stable electrode potential after each increment, and plot on semi-logarithmic paper the concentration (log axis) against the potential (linear axis). See [Figure 1](#) Prepare new calibration curve with fresh standard each day.

5. Rinse electrodes in electrode rinse solution, blot dry, and place in sample. Add 1ml low-level ISA to 100ml sample. Adjust pH above 9, if necessary.
6. Place electrodes in sample. Stir thoroughly. Wait for a stable reading and record.
7. Determine sample concentration corresponding to the measured potential from the low-level calibration curve.

Calibration Curve for Low-level Measurement.

Additions of 100ppm or 10^{-3} M standard to 100ml distilled water, plus 1ml low-level ISA. A is a 1ml graduated pipette. B is a 2ml pipette.

Step	Pipette	Added Volume	Concentration ppm	Molarity
1	A	0.1 ml	0.10	1.0×10^{-6}
2	A	0.1	0.20	2.0×10^{-6}
3	A	0.2	0.40	4.0×10^{-6}
4	A	0.2	0.60	6.0×10^{-6}
5	A	0.4	0.99	9.9×10^{-6}
6	B	2.0	2.91	2.9×10^{-5}
7	B	2.0	4.76	4.8×10^{-5}

Troubleshooting Checklist

<u>Symptom</u>	<u>Possible Causes</u>	<u>Next Step</u>
Off-Scale or short- Over Range Reading meter instr-	Defective meter	Check meter with ing strap (see uction manual)
and	Electrodes not plugged in properly	Unplug electrodes reset
lift outer a few solution	Reference electrode junction is dry	Hold cap and sleeve to expel drops of filling
	Reference electrode not filled	Be sure reference electrode is filled
Meter cannot be Calibrated	Calibration control not turned far enough	Continue turning the calibration control
Noisy or unstable Readings (readings changing randomly)	Defective meter	Check meter with shorting strap
ISA	ISA not used	Use recommended
or	Meter or stirrer not grounded	Ground meter stirrer
Drift (reading slowly to come	Samples and standards	Allow solutions

Changing in one temperature	at different temperatures	to room
		Before measurement
recommended	Incorrect reference	Use
	filling solution	filling solution
solution	Membrane hydrated or Exposed to interferences	Soak overnight in electrode storage
	pH too low	Adjust pH
ISA	ISA not used	Use recommended

Low Slope or standards	Standards contaminated	Prepare fresh
No Slope	or incorrectly made	standards
	ISA not used	Use recommended
ISA	Standard used as ISA	Use ISA
	pH too low	Adjust pH
solution	Membrane hydrated or Exposed to interferences	Soak overnight in electrode storage

"Wrong Answer" on the	Incorrect scaling of	Plot millivolts
-----------------------	----------------------	-----------------

(but calibration On the log curve is "ok") concentration decade concentrations.	semilog paper	the linear axis. axis, be sure numbers within each are increasing with increasing
sign of correctly	Incorrect sign	Be sure to note millivolt reading
standards	Incorrect standards	Prepare fresh
conversion 23.0ppm	Wrong units	Apply correct factor: $10^{-3}\text{M} =$
	Sample pH too low	Adjust pH above 9

Known Addition

Known addition is convenient for measuring occasional samples because no calibration curve is needed. Since an accurate measurement requires that the concentration double because of the addition, sample concentration must be known to within a factor of three. Known addition is a convenient check on the results of direct measurement.

Using Digital pH/mV Meter

1. To measure an unknown sample, place electrodes in 100ml sample and add 2ml ISA. Stir thoroughly.
2. Turn the function switch of the meter to mV. When the reading is stable, set the meter reading to 000.0 by turning the Calibration Control. if the reading cannot be set to 000.0, record the potential reading.
3. Prepare a standard solution about 10 times as concentrated as the sample concentration by diluting 0.1M standard or the 1000ppm stock solution. Add 2ml ISA to each 100ml standard.
4. Pipette 10ml standard into the sample. Stir thoroughly. When the reading is stable, record the potential difference, E, directly from the meter. If the meter could not be zeroed in step 2, subtract the first reading from the second to find E.
5. From Table 3 find the value, Q, that corresponds to the change in potential, E. To determine the original sample concentration, multiply Q by the concentration of the added standard:

$$C_0 = QC_s$$

Where

C_0 = Sample Concentration

Q = Reading from known addition table

C_s = Concentration of added standard

The table of Q values is calculated for a 10% volume change for electrodes with a slope of 59mV. The equation for the calculation of Q for different slopes and volume changes is given below.

$$Q = \frac{1}{(1 + p) 10^{\Delta E/S - 1}}$$

where

$$P = \frac{\text{Volume of Standard}}{\text{Volume of Slope}}$$

$$S = \text{Slope}$$

Table 3

Known Addition Table

Values for Q vs. ΔE at 25°C

(59 mV Slope) for 10% volume change

ΔE	Q	ΔE	Q	ΔE	Q	ΔE	Q
5.0	0.297	10.0	0.160	20.0	0.0716	30.0	0.0394
5.1	0.293	10.2	0.157	20.2	0.0707	30.2	0.0390
5.2	0.288	10.4	0.154	20.4	0.0698	30.4	0.0386
5.3	0.284	10.6	0.151	20.6	0.0689	30.6	0.0382
5.4	0.280	10.8	0.148	20.8	0.0680	30.8	0.0378
5.5	0.276	11.0	0.145	21.0	0.0671	31.0	0.0374
5.6	0.272	11.2	0.143	21.2	0.0662	31.2	0.0370
5.7	0.268	11.4	0.140	21.4	0.0654	31.4	0.0366
5.8	0.264	11.6	0.137	21.6	0.0645	31.6	0.0362
5.9	0.260	11.8	0.135	21.8	0.0637	31.8	0.0358
6.0	0.257	12.0	0.133	22.0	0.0629	32.0	0.0354
6.1	0.253	12.2	0.130	22.2	0.0621	32.2	0.0351
6.2	0.250	12.4	0.128	22.4	0.0613	32.4	0.0347
6.3	0.247	12.6	0.126	22.6	0.0606	32.6	0.0343
6.4	0.243	12.8	0.123	22.8	0.0598	32.8	0.0340
6.5	0.240	13.0	0.121	23.0	0.0591	33.0	0.0336
6.6	0.237	13.2	0.119	23.2	0.0584	33.2	0.0333
6.7	0.234	13.4	0.117	23.4	0.0576	33.4	0.0329
6.8	0.231	13.6	0.115	23.6	0.0569	33.6	0.0326
6.9	0.228	13.8	0.113	23.8	0.0563	33.8	0.0323

7.0	0.225	14.0	0.112	24.0	0.0556	34.0	0.0319
7.1	0.222	14.2	0.110	24.2	0.0549	34.2	0.0316
7.2	0.219	14.4	0.108	24.4	0.0543	34.4	0.0313
7.3	0.217	14.6	0.106	24.6	0.0536	34.6	0.0310
7.4	0.214	14.8	0.105	24.8	0.0530	34.8	0.0307
7.5	0.212	15.0	0.103	25.0	0.0523	35.0	0.0304
7.6	0.209	15.2	0.1013	25.2	0.0517	36.0	0.0289
7.7	0.207	15.4	0.0997	25.4	0.0511	37.0	0.0275
7.8	0.204	15.6	0.0982	25.6	0.0505	38.0	0.0261
7.9	0.202	15.8	0.0967	25.8	0.0499	39.0	0.0249
8.0	0.199	16.0	0.0952	26.0	0.0494	40.0	0.0237
8.1	0.197	16.2	0.0938	26.2	0.0488	41.0	0.0226
8.2	0.195	16.4	0.0294	26.4	0.0482	42.0	0.0216
8.3	0.193	16.6	0.0910	26.6	0.0477	43.0	0.0206
8.4	0.190	16.8	0.0897	26.8	0.0471	44.0	0.0196
8.5	0.188	17.0	0.0884	27.0	0.0466	45.0	0.0187
8.6	0.186	17.2	0.0871	27.2	0.0461	46.0	0.0179
8.7	0.184	17.4	0.0858	27.4	0.0456	47.0	0.0171
8.8	0.182	17.6	0.0846	27.6	0.0450	48.0	0.0163
8.9	0.180	17.8	0.0834	27.8	0.0445	49.0	0.0156

Continued.....

9.0	0.178	18.0	0.0822	28.0	0.0440
50.0	0.0149				
9.1	0.176	18.2	0.0811	28.2	0.0435
51.0	0.0143				
9.2	0.174	18.4	0.0799	28.4	0.0431
52.0	0.0137				
9.3	0.173	18.6	0.0788	28.6	0.0426
53.0	0.0131				
9.4	0.171	18.8	0.0777	28.8	0.0421
54.0	0.0125				
9.5	0.169	19.0	0.0767	29.0	0.0417
55.0	0.0120				
9.6	0.167	19.2	0.0756	29.2	0.0412
56.0	0.0115				
9.7	0.165	19.4	0.0746	29.4	0.0408
57.0	0.0110				
9.8	0.164	19.6	0.0736	29.6	0.0403
58.0	0.0105				

9.9	0.162	19.8	0.0726	29.8	0.0399
59.0	0.0101				

Measuring Hints

- Between measurements, store electrodes in electrode storage solution. DO NOT STORE IN WATER OR AIR. For low-level measurements, a more dilute NaCl solution with pH adjusted by adding ISA may be used as a storage media. New electrodes should be placed overnight in the storage solution prior to use.
- For maximum precision, allow all standards and samples to come to ambient temperature before measurement. Do not use electrode above 60°C.
- Stir samples and standards during measurement. For best results stir at a rate that will not cause a vortex. Magnetic stirrers are recommended, but some models generate sufficient heat to change solution temperature. Place a piece of insulating material such as cork, cardboard, or styrofoam between the stirrer and heater.
- Rinse the electrodes between measurements to prevent solution carryover. Use a rinse bottle filled with electrode rinse solution.
- Make all measurements in basic solutions. Adjust all samples and standards to pH > 9 with ISA.
- Use plastic laboratory ware for low-level measurements.
- DO NOT etch electrode with ammonium bi-fluoride, NH_4F -HF, under any circumstances.

Electrode Characteristics

Electrode Response

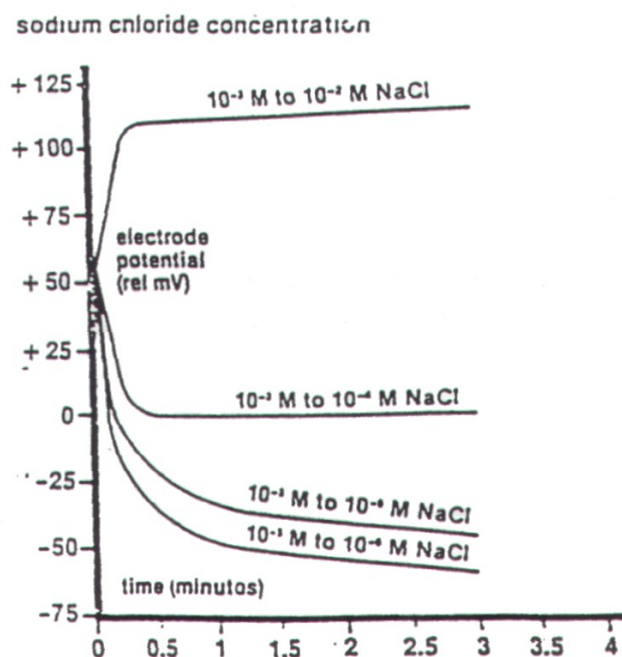
The electrode potential plotted against concentration on semi-logarithmic paper results in a straight line with a slope of about 57mV per decade. See Figure 1.

The electrode exhibits good time response (99% response in one minute or less) for concentrations above 10^{-5} M. Below this value response times are considerably longer. See Figure 2.

After prolonged use a hydrated layer forms on the surface of the sodium-sensitive glass, which gives rise to increased response times. This layer may be treated with electrode storage solution, to restore normal performance. See measuring hints.

Figure 2

Typical Electrode Response to Step Changes in Sodium Chloride Concentrations.



Reproducibility

Reproducibility is limited by factors such as temperature fluctuations, drift and noise. Within the electrode's operating range, reproducibility is independent of concentration. With calibration every hour, direct electrode measurements reproducible to $\pm 2\%$ can be obtained.

Interference's

Some other cations, if present at high enough levels, are electrode interference's and will cause measurement errors or electrode drift.

Table 4 indicates levels of common cations that will cause a 10% error at 10^{-4} , 10^{-3} and 10^{-2} M or at 1, 10, and 100ppm Sodium.

In practically all samples, most cations listed in table 4 are absent or insignificantly low. Using ammonium ion in the recommended ISA does not result in an error, provided that all standards and samples have the same level of ISA added.

In cases where interference's are high, the electrode may become drifty and sluggish in response. When this happens, soak electrode in electrode storage solution. See measuring hints.

Table 4

Levels of Possible Interference's Causing a 10% Error at Various Levels of Sodium.

Interference's

Moles per Litre	10^{-4} M Na^+	10^{-3} M Na^+	10^{-2} M Na^+
Li^+	5×10^{-4} M	5×10^{-3} M	5×10^{-2} M
K^+	1×10^{-2}	0.1	1
Rb^+	0.3	3	-
NH_4^+	0.3	3	-
Ag^+ 10^{-7}	3×10^{-9}	3×10^{-8}	3×10^{-7}
Tl^+	5×10^{-2}	0.5	-

Interference's

ppm	1ppm Na^+	10ppm Na^+	100ppm Na^+
Li^+	2ppm	15ppm	150ppm
K^+	170	1,700	17,000
Rb^+	11,000	110,000	-
NH_4^+ as N	1,800	18,000	-
Ag^+	10^{-4}	10^{-3}	10^{-2}
Tl^+	4,5000	45,000	-

Limit of Detection

The free sodium concentration in basic solutions can be measured down to 10^{-6} M. Measurements below 10^{-5} M Sodium must be made with extreme care as significant pickup of sodium ion may occur due to desorption from container walls. Ordinary glass will dissolve sufficiently to produce spurious results at low levels. Plastic laboratory ware is recommended.

Temperature Effects

Changes in temperature will cause the electrode response curve to both shift and change slope. Table 5 indicates the variation of theoretical slope with temperature. Samples and standards should be at the same temperature: for convenience, room temperature.

The electrode can be used at temperatures from -5°C to 70°C , provided that temperatures equilibrium has occurred. For use at temperatures substantially different from room temperature, equilibrium times up to an hour are recommended. It is not advisable to use the electrode above 70°C , since electrode response may be permanently altered.

Table 5

Values of Electrode Slope vs. Temperature

T° C	S	T° C	S
0	54.20	30	60.15
10	56.18	40	62.13
20	58.16	50	64.11

25

59.16

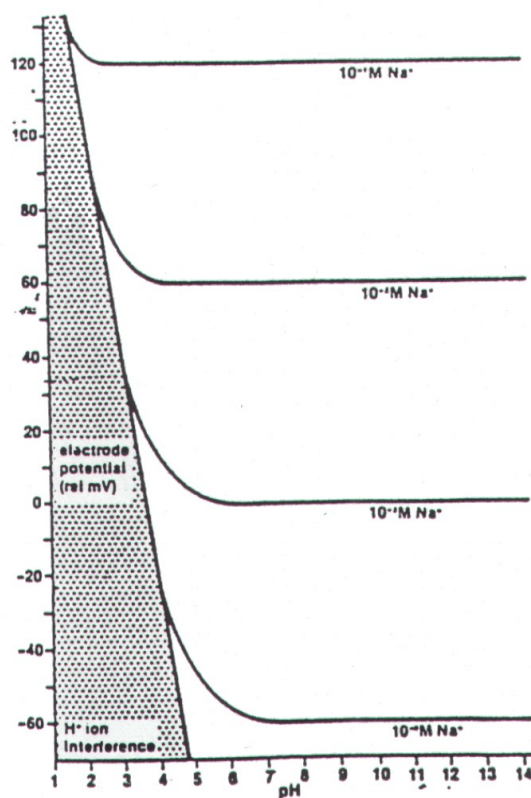
pH Effects

The electrode response to sodium ion in solutions at various pH is shown in figure 3. Although the electrode can be used over a wide pH range, hydrogen ion interferes with measurements of low levels of sodium ion. The edge of the shaded area to the left in figure 3 indicates a minimum pH at which dilute sodium measurements can be made with less than 10% hydrogen ion interference.

For optimum results over the entire concentration range of sodium, the pH should be adjusted to greater than 9 by the addition of ISA to all samples and standards. If necessary, additional ammonium hydroxide treatment will help adjust the pH to the desired pH level in problematic samples.

Figure 3

Sodium Electrode Potential Behaviour vs. Solution pH in Pure NaCl Solution at 25°C



Electrode Storage

The 315-75 or 315-75-C should be stored in the electrode storage solution described. Under no circumstances should either electrode be stored in distilled water or air. Before low-level measurements, a more dilute NaCl solution with pH adjusted by adding ISA may be used for storage.

Theory of Operation

The sodium electrode consists of a sodium-containing glass membrane. When the membrane is in contact with a sodium solution, a difference in electrode potential is measured against a constant reference potential with a digital pH/mV meter or specific ion meter. The measured potential corresponding to the level of the sodium ion in solution is described by the nernst equation:

$$E = E_0 + S \log (A)$$

Where:

E = Measured electrode potential

E₀ = Reference potential (a constant)

A = Sodium ion level in solution

S = Electrode slope (about 57mV per decade)

The level of sodium ion in solution, A, is the activity or "effective concentration". The sodium activity is related to the sodium ion concentration, C, by the ionic activity coefficient, Y:

$$A = yC$$

Ionic activity coefficients are variable and largely depend on total ionic strength.

Ionic strength is defined as:

$$\text{ionic strength} = \frac{1}{2} \sum C_i Z_i^2$$

where:

C_i = Concentration of ion i

Z_i = Charge of ion i

If background ionic strength is high and constant relative to the sensed ion concentration, the activity coefficient is constant and activity is directly proportional to concentration.

Ionic strength adjustor (ISA) is added to all sodium standards and samples so that the background ionic strength is high and constant relative to variable concentrations of sodium ion. For the sodium electrode, an ammonium chloride-ammonia buffer is the recommended ISA. Other solutions, properly adjusted for pH, can be used as long as they do not contain ions that would interfere with the electrode's response to sodium ion.

Reference electrode condition must also be considered. Liquid junction potentials arise any time two solutions of different composition are brought into contact. The potential results from the interdiffusion of ions in the two solutions. Since ions diffuse at different rates, electrode change will be carried unequally across the solution boundary resulting in a potential difference between the two solutions. In making electrode measurement it is important that this potential be the same when the reference is in the standardising solution as well as in the sample solution; otherwise, the change in liquid junction potential will appear as an error in the measured specific ion electrode potential.

The most important variable which the analyst has under his control is the composition of the liquid junction filling solution. The filling solution should be equitransferent. That is, the speed with which the positive and negative ions in the filling solution diffuse into the sample should be as nearly equal as possible. If the rate at which positive and negative charge is carried into the sample solution is equal, then no junction potential can result.

However, there are a few samples where no filling solution adequately fulfils the condition stated above. Particularly troublesome are samples containing high levels of strong acids (pH 0-2) or strong bases (pH 12-14). The high mobility of hydrogen and hydroxide ions in samples makes it impossible to "swamp out" their effect on the junction potential with any concentration of an equitransferant salt. For these solutions it is recommended to: calibrate in the same pH range as the sample; or, use a known increment method for ion measurement.

Specifications

<u>Concentration</u>	Saturated to 10^{-6} M
<u>Range</u>	(0.02 ppm)
<u>pH Range</u>	3 to 12 pH (depending on Na^+ level)
<u>Temperature Range</u>	-5°C to 70°C
<u>Electrode Resistance</u>	Less than 200 megohms
<u>Reproducibility</u>	$\pm 2\%$
<u>Storage</u>	Store in 1ppm Sodium Solution
<u>Size</u>	Length: 120 mm Diameter: 1.2cm

Cap diameter: 1.5cm

Cable length: 100cm

Specifications subject to change without notice.