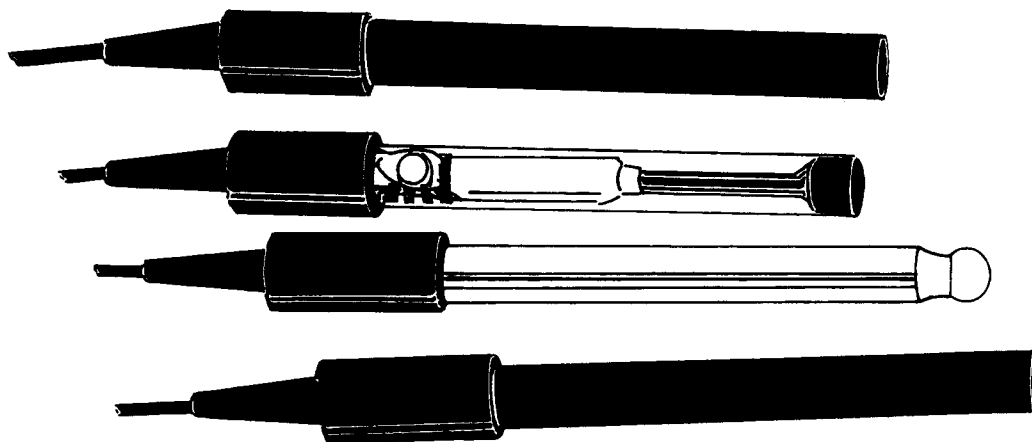


 **ISE-8745**

 **Potassium Ion Selective Electrodes**



Operator's Manual
M781/1092

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SECTION 1 GENERAL DESCRIPTION

The OMEGA® Potassium Ion Electrodes are used to quickly, simply, accurately, and economically measure potassium ions in aqueous solutions.

SECTION 2 PREPARATION FOR MEASUREMENT

2.1 REQUIRED EQUIPMENT

1. A pH/mV meter or an ion meter, either line operated or portable.
2. Semi-logarithmic 4-cycle graph paper for preparing calibration curves when using the meter in the mV mode.
3. A magnetic stirrer.
4. The Potassium Ion Electrode, ISE-8745 (reference electrode necessary).
5. The Double Junction Reference Electrode, PHE-3211, in the inner junction and with 1M NaCl in the outer junction.

2.2 REQUIRED SOLUTIONS

1. Deionized or distilled water for solution and standard solution.
2. ISA (ionic strength adjuster) to adjust the pH and to keep a constant background ionic strength present in the solution. To prepare the 5M NaCl ISA, add 29.2 grams of reagent-grade Sodium Chloride (NaCl) to a 100 mL volumetric flask about half full of distilled water to the mark, cap, and invert the flask several times to mix. Add 2 mL of ISA to every 100 mL of sample or standard solution for an ionic background strength of 0.10M.
3. Potassium Standard, 0.1M KCl, ISE-8745-S1
To prepare this standard, add 7.46 grams of reagent-grade potassium chloride to a 1 liter volumetric flask about half full of distilled water. Swirl the flask to dissolve the solid and fill to the mark with distilled water. Cap the flask and invert several times to mix the solution.
4. 1000ppm Potassium Standard. To prepare this standard, add 256 mL of the 0.1M standard to a 1 liter volumetric and dilute to the mark with distilled water. Cap the flask and invert several times to mix the solution.

2.3 ELECTRODE PREPARATION

Remove the rubber caps covering the electrode tip and the rubber bands covering the filling holes of the reference electrode. Fill the combination electrode or the reference electrode salt bridge with the appropriate filling solution.

Connect the electrodes to the proper terminals as recommended by the meter manufacturer.

NOTE

Do not be alarmed if white crystals form at end of electrode. This is normal with pH electrodes. These crystals are potassium chloride. Remove the shipping cap and rinse with distilled water to dissolve the crystals.

2.3.1 Electrode Slope Check (with standard pH/mV meter)

The electrodes should be checked on a daily basis. To a 150 mL beaker, add 100 mL of distilled water and 2 mL of ISA. Place the beaker on a magnetic stirrer and begin stirring at a constant rate. After assuring that the meter is in the mV mode, lower the electrode tip into the solution. If drifting or instability is observed, see the Troubleshooting Section.

Using a pipet, add 1 mL of 0.1 M or 1000ppm potassium standard to the beaker. When the reading is stable, record the millivolt reading.

Using a pipet, add 10 mL of the 0.1M or 1000 potassium standard to the beaker. When the reading has stabilized, record the millivolt reading.

Determine the difference between the two readings. A difference of 58 ± 2 mV indicates correct electrode operation, assuming the solution temperature is between 20° and 25°C. See the Troubleshooting Section if the potential change is not within this range.

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

2.3.2 Electrode Slope Check (with ion meter)

Prepare standard potassium solutions whose concentrations vary by tenfold. Use either the 0.1M KCl or the 1000 ppm K^{+1} standard stock solutions. Use the serial dilution method for this preparation.

To a 150 mL beaker, add 100 mL of the lower value standard and 2 mL of ISA. Place the beaker on the magnetic stirrer and begin stirring at a constant rate. Lower the electrode tip into the solution.

Assure that the meter is in the concentration mode. Adjust the meter to the concentration of the standard and fix the value in the memory according to the meter manufacturer's instructions.

Rinse the electrode with distilled water and blot dry.

To a 150 mL beaker, add 100 mL of the higher value standard and 2 mL of ISA. Place the beaker on the magnetic stirrer and begin stirring at a constant rate. Lower the electrode tip into the solution.

Adjust the meter to the concentration of the standard and fix the value in the memory.

Read the electrode slope according to the meter manufacturer's instructions. Correct electrode operation is indicated by a slope of 90-100%. See the Troubleshooting Section if the slope is not within this range.

SECTION 3 MEASUREMENT PROCEDURE

3.1 MEASURING HINTS

All samples and standards should be at the same temperature for precise measurement, preferably ambient temperature.

Constant, but not violent, stirring is necessary for accurate measurement. Magnetic stirrers can generate sufficient heat to change the solution temperature. To counteract this effect, place a piece of insulating material, such as styrofoam sheet or asbestos sheet, between the stirrer and beaker.

Always rinse the electrode tip with distilled water and blot dry between readings to prevent solution carryover.

Check the electrode for air bubbles adhering to the membrane surface after immersion in solution. Agitate the electrode gently to remove the air bubbles.

A slow or sluggish electrode response may indicate surface contamination of the membrane. Soak the electrode tip in distilled water for about 5 minutes to clean the membrane. Rinse the membrane and soak in diluted standard solution for about 5 minutes to restore performance.

When measuring samples with high ionic strength, prepare standards with compositions similar to that of the sample. Dilute concentrated samples (over 0.1M) before measurement.

3.2 SAMPLE REQUIREMENTS

Make sure that the samples and standards are at the same temperature. About a 2% error will be introduced for a 1°C difference in temperature. Temperature should normally be less than 40°C with intermittent measurements allowed to 50°F.

All samples and standards must be aqueous. They must not contain organic solvents.

Interferences found in Table 4-1 should be absent.

3.3 UNITS OF MEASUREMENT

Potassium concentrations are measured in units of parts per million as potassium, parts per million as KCl, moles per liter, or any other convenient concentration unit. Table 3-1 indicates some of the concentration units.

TABLE 3-1
Concentration Unit Conversion Factors

<u>ppm K⁺¹</u>	<u>ppm KCl</u>	<u>moles/liter K⁺¹</u>
3.91	7.46	1 x 10 ⁻⁴
39.1	74.6	1 x 10 ⁻³
391	746	1 x 10 ⁻²

3.4 DIRECT MEASUREMENT (using standard pH/mV meter)

Direct measurement is a simple procedure for measuring a large number of samples. A single meter reading is all that is required for each sample. The ionic strength of samples and standards should be made the same by adjustment with ISA for all potassium samples. The temperature of both sample solution and of standard solutions should be the same.

1. By serial dilution, prepare 10⁻², 10⁻³, and 10⁻⁴M or 100, 10, and 1ppm standards by serial dilution of the 0.1M or 1000 ppm standard. Add 2 mL of ISA per 100 mL of standard.
2. Place the most dilute solution (10⁻⁴M or 1 ppm) on the magnetic stirrer and begin stirring at a constant rate. After assuring that the meter is in the mV mode, lower the electrode tip into the solution. When the reading has stabilized, record the mV reading.
3. Place the midrange solution (10⁻³M or 10 ppm) on the magnetic stirrer and begin stirring. After rinsing the electrode with distilled water and blotting dry, immerse the electrode tip in the solution. When the reading has stabilized, record the mV reading.
4. Place the most concentrated solution (10⁻²M or 100 ppm) on the magnetic stirrer and begin stirring. After rinsing the electrode with distilled water and blotting dry, immerse the electrode tip in the solution. When the reading has stabilized, record the mV reading.
5. Using the semi-logarithmic graph paper, plot the mV reading (linear axis) against the concentration (log axis). A typical calibration curve can be found in Figure 3-1.

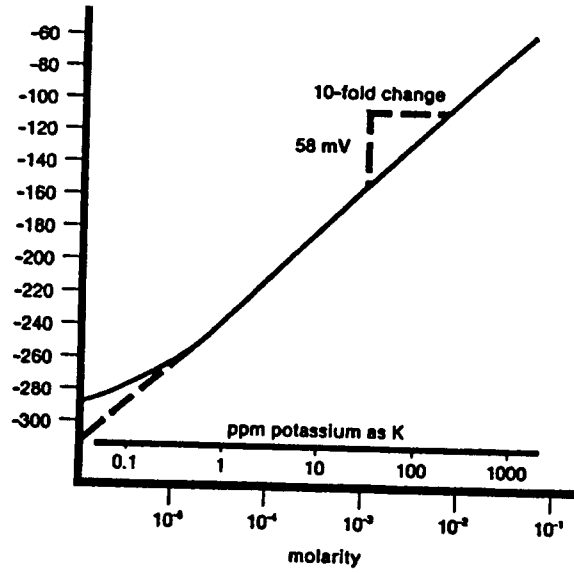


Figure 3-1 Typical Potassium Electrode Calibration Curve

A calibration curve is constructed on semi-logarithmic paper when using a pH/mV meter in the direct measurement procedure. The measured electrode potential in mV (linear axis) is plotted against the standard concentration (log axis). In the linear region of the curve, only three standards are necessary to determine a calibration curve. In the non-linear region, additional points must be measured. The direct measurement procedures given are for the linear portion of the curve. The non-linear portion of the curve requires the use of low level procedures.

Extrapolate the curve down to about $1 \times 10^{-5} \text{M}$ or 0.4 ppm.

6. To a clean, dry 150 mL beaker, add 100 mL of sample and 2 mL of ISA. Place the beaker on the magnetic stirrer and begin stirring. Place the electrode tip in the solution. When the reading has stabilized, record the millivolt reading. Determine the concentration directly from the calibration curve.
7. The electrode should be re-calibrated every 1-2 hours. Simply repeat Steps 2-5.

3.5 DIRECT MEASUREMENT (using an ion meter)

1. By serial dilution of the 0.1M or 1000 ppm potassium standard, prepare two potassium standards whose concentration is near the expected sample concentration. Measure out 100 mL of each standard into individual 150 mL beakers and add 2 mL of ISA to each.
2. Place the most dilute solution on the magnetic stirrer and begin stirring at a constant rate. Assure that the meter is in the concentration mode.
3. Lower the electrode tip into the solution.
4. Adjust the meter to the concentration of the potassium standard and fix the value in the memory according to the meter manufacturer's instructions after stabilization of the reading.
5. Rinse the electrode tip with distilled water and blot dry.
6. Place the most concentrated solution on the magnetic stirrer and begin stirring at a constant rate.
7. Lower the electrode tip into the solution.
8. Adjust the meter to the concentration of the potassium standard and fix the value in the memory according to the meter manufacturer's instructions after stabilization of the reading.

9. For low level measurements, place the rinsed, dried electrode into a solution containing 100 mL of distilled water and 2 mL of ISA. After stabilization, fix the blank value in the meter according to the meter manufacturer's instructions.
10. Place 100 mL of the sample and 2 mL of ISA in a 150 mL beaker, place it on the magnetic stirrer, and begin stirring.
11. Immerse the electrode tip in the solution and wait for the reading to stabilize. Read the concentration directly from the meter display.
12. The electrodes should be re-calibrated every 1-2 hours. Simply repeat Steps 2-8 (2-9).

3.6 LOW LEVEL POTASSIUM DETERMINATION

This procedure is recommended for solutions with ionic strengths less than 1.0×10^{-2} M. If the solution is high in ionic strength, but low in potassium, use the same procedure, but prepare a calibration solution with a composition similar to the sample.

1. Using 20 mL of stock ISA, dilute to 100 mL with distilled water.
2. Dilute 1 mL of the 0.1M standard to 100 mL to prepare a 1.0×10^{-3} M standard solution for measurements in moles per liter. Dilute 10 mL of the 1000 ppm standard to prepare a 100 ppm standard solution for measurements in ppm.
3. To a 150 mL beaker, add 100 mL of distilled water and 1 mL of low level ISA. Place the beaker on the magnetic stirrer and begin stirring at a constant rate.
4. Place the electrode tip in the solution. Assure that the meter is in the mV mode.
5. Add increments of the 1.0×10^{-3} M or 100 ppm standard as given in Table 3-2 below.
6. After the reading has stabilized, record the mV reading after each addition.

TABLE 3-2
STEPWISE CALIBRATION FOR LOW LEVEL POTASSIUM MEASUREMENTS

STEP	PIPET	ADDED CONCENTRATION		
		VOLUME (mL)	M	ppm
1	A	0.1	1.0×10^{-6}	0.1
2	A	0.1	2.0×10^{-6}	0.2
3	A	0.2	4.0×10^{-6}	0.4
4	A	0.2	6.0×10^{-6}	0.6
5	A	0.4	9.9×10^{-6}	1.0
6	B	2	2.9×10^{-5}	2.9
7	B	2	4.8×10^{-5}	4.8

Pipet A = 1 mL graduated pipet

Pipet B = 2 mL pipet

Solutions: additions of 1.0×10^{-3} M or 100 ppm standard to 100 mL of distilled water and 1 mL of low level ISA.

7. On semi-logarithmic graph paper, plot the mV reading (linear axis) against the concentration (log axis) as in Figure 3-1.
8. Rinse the electrode in distilled water and blot dry.
9. Measure out 100 mL of the sample into a 150 mL beaker, add 1 mL of low level ISA, and place the beaker on the magnetic stirrer. Begin stirring.
10. After the reading has stabilized, record the mV reading and determine the concentration from the low level calibration curve.
11. Prepare a new low level calibration curve daily. Check the calibration curve every 1-2 hours by repeating Steps 3-7 above.

For low level potassium determination using an ion meter, follow the procedure given for normal potassium determinations using an ion meter and the blank correction procedure.

SECTION 4 ELECTRODE CHARACTERISTICS

4.1 REPRODUCIBILITY

Direct electrode measurements reproducible to 2% can be obtained if the electrode is calibrated every hour. Factors such as temperature fluctuations, drift, and noise limit reproducibility. Reproducibility is independent of concentration within the electrode's operating range.

4.2 INTERFERENCES

Table 4-1 lists some common cations that, if present in high enough levels, will cause electrode interferences and measurement errors or electrode drift when using the potassium ion electrodes.

Electrode drift and slow response could indicate the presence of high interference from the ions listed. Soak the electrode in distilled water for an hour, then for two hours in potassium standard solution to restore proper response.

TABLE 4-1
Concentration of Possible Interferences Causing a 10% Error at Various Levels of KCl; Background Ionic Strength is 0.12M NaCl

<u>INTERFERENCES</u> <u>(MOLES/LITER)</u>	<u>10⁻²M K⁺¹</u>	<u>10⁻³M K⁺¹</u>	<u>10⁻⁴M K⁺¹</u>
Cs ⁺¹	3.0X10 ⁻³	3.0X10 ⁻⁴	3.0X10 ⁻⁵
NH ₄ ⁺¹	6.0X10 ⁻²	6.0X10 ⁻³	6.0X10 ⁻⁴
Tl ⁺¹	6.0X10 ⁻²	6.0X10 ⁻³	6.0X10 ⁻⁴
H ⁺¹	1.0x10 ⁻¹	1.0X10 ⁻²	1.0X10 ⁻³
Ag ⁺¹	1.0x10 ¹	1.0	1.0x10 ⁻¹
+Tris ⁺¹	1.0x10 ¹	1.0	1.0x10 ⁻¹
Li ⁺¹	2.0x10 ¹	2.0	2.0X10 ⁻¹
Na ⁺¹	2.0X10 ¹	2.0	2.0X10 ⁻¹

+Tris⁺¹ is the cation of tris (hydroxymethyl) aminomethane.

<u>INTERFERENCES</u> <u>(ppm)</u>	<u>100 ppm K⁺¹</u>	<u>10 ppm K⁺¹</u>	<u>1 ppm K⁺¹</u>
Cs ⁺¹	1.0x10 ²	1.0x10 ¹	1.0
NH ₄ ⁺¹	2.7X10 ²	2.7X10 ¹	2.7
Tl ⁺¹	3.14X10 ³	3.14X10 ²	3.14X10 ¹
H ⁺¹	1.6pH	2.6pH	3.6pH
Ag ⁺¹	2.765x10 ⁵	2.765x10 ⁴	2.765x10 ³
+Tris ⁺¹	3.105X10 ⁵	3.105X10 ⁴	3.105X10 ³
Li ⁺¹	3.56X10 ⁴	3.56X10 ³	3.56X10 ²
Na ⁺¹	1.179X10 ⁵	1.179X10 ⁴	1.179X10 ³

4.3 TEMPERATURE INFLUENCES

Samples and standards should be at the same temperature, since electrode potentials are influenced by changes in temperature. A 1°C difference in temperature results in a 2% error at the 1.0x10⁻³M level.

Provided that temperature equilibria has occurred, the potassium ion electrodes can be used at other than room temperature, however, measurements at temperatures markedly different from room temperature may require equilibrium times up to one hour. Table 4-2 indicates the variation of theoretical slope with temperature.

TABLE 4-2
Temperature vs. Values for the Electrode Slope

<u>Temp. (°C)</u>	<u>"S" (slope)</u>
0	54.20
10	56.18
20	58.16
25	59.16
30	60.15
40	62.13
50	64.11

4.4 ELECTRODE RESPONSE

Plotting the electrode mV potential against the potassium concentration on semi-logarithmic paper results in a straight line with a slope of about 58mV per decade. Refer to Figure 3-1.

The time needed to reach 99% of the stable electrode potential reading, the electrode response time, varies from one minute or less for potassium concentration above $1.0 \times 10^{-5} M$ to several minutes near the detection limit. Refer to Figure 4-1.

4.5 ELECTRODE STORAGE

The Sodium Ion Electrodes may be stored in $1.0 \times 10^{-2} M$ potassium standard for short periods of time. For storage over 3 weeks, rinse and dry the indicating electrode and cover the tip with the rubber cap. The reference portion of the combination electrode (or the salt-bridge of the reference electrode) should be drained of filling solution, the rubber band should be placed over the filling hole and the rubber cap replaced on the electrode tip.

4.6 LIMITS OF DETECTION

The upper limit of detection is 1M in pure potassium chloride solutions. The upper limit of detection is above $1.0 \times 10^{-1} M$ when other ions are present, but the possibility of a liquid junction potential developing at the reference electrode and the "salt extraction effect" are two limiting factors. Some salts may be extracted into the electrode membrane at high salt concentrations causing deviation from theoretical response. Calibrate the electrode at four or five intermediate points, or dilute the sample, to measure samples between $1.0 \times 10^{-1} M$ and 1M.

The slight water solubility of the ion exchanger in the sensing module, which causes deviation from theoretical response, determines the lower limit of detection. The theoretical response at low levels of potassium chloride compared to actual response is shown in Figure 3-1. A low level measurement is recommended if potassium measurements are made below $1.0 \times 10^{-5} \text{M}$ (0.39 ppm as potassium).

4.7 pH EFFECTS

Hydrogen ion interferes with measurements of low levels of potassium ion although the electrode can be used over a wide pH range. Table 4-1 should be used to determine the minimum pH of which low level potassium measurements can be made without more than a 10% error due to hydrogen ion interference.

4.8 THEORY OF OPERATION

The potassium electrode consists of an electrode body containing an ion exchanger in a sensing module. This sensing module contains a liquid internal filling solution in contact with a gelled organophilic membrane containing a potassium selective ion exchanger.

An electrode potential develops across the membrane when the membrane is in contact with a potassium solution. Measurement of this potential against a constant reference potential with a digital pH/mV meter or with a specific ion meter depends on the level of free potassium ion in solution. The level of potassium ions, corresponding to the measured potential, is described by the Nernst Equation:

$$E = E_0 + \text{Slope } X$$

Where:

E = measured electrode potential

E_0 = reference potential (a constant)

S = electrode slope (-58 mV/decade)

X = level of potassium ions in solution

The activity, X, represents the effective concentration of the ions in solution total potassium concentration, C_t includes free potassium ions, C_f plus bound or complexed potassium ions, C_b . Since the potassium electrodes only respond to free ion, the free ion concentration is:

$$C_f = C_t - C_b$$

The activity is related to the free ion concentration, C_f , by the activity coefficient, γ by:

$$X = C_f \gamma$$

Activity coefficients vary, depending on total ionic strength, I , defined as:

$$I = 1/2 \sum C_x Z_x^2$$

Where:

C_x = concentration of ion X

Z_x = charge of ion X

Σ = Sum of all of the types of ions in the solution.

In the case of high and constant ionic strength relative to the sensed ion concentration, the activity coefficient, γ , is constant and the activity, X , is directly proportional to the concentration.

To adjust the background ionic strength to a high and constant value, ionic strength adjustor (ISA) is added to samples and standards. The recommended ISA for potassium is sodium chloride, NaCl. Solutions other than this may be used as long as the ions that they contain do not interfere with the electrode's response to potassium ions.

The reference electrode must also be considered. When two solutions of different composition are brought into contact with one another, liquid junction potentials arise. Millivolt potentials occur from the interdiffusion of ions into the two solutions. Electrode charge will be carried unequally across the solution boundary resulting in a potential difference between the two solutions, since ions diffuse at different rates. When making measurements, it is important to remember that this potential be the same when the reference is in the standardizing solution as well as in the sample solution or the change in liquid junction potential will appear as an error in the measured electrode potential.

The composition of the liquid junction filling solution in the reference electrode is most important. The speed with which the positive and negative ions in the filling solution diffuse into the sample should be as nearly equal as possible, that is, the filling solution should be equitransferent. No junction potential can result if the rate at which positive and negative charge carried into the sample are equal.

Strongly acidic (pH = 0-2) and strongly basic (pH = 12-14) solutions are particularly troublesome to measure. The high mobility of hydrogen and hydroxide ions in samples make it impossible to mask their effect on the junction potential with any concentration of an equitransferent salt. One must either calibrate the electrodes in the same pH range as the samples or use a known increment method for ion measurement.

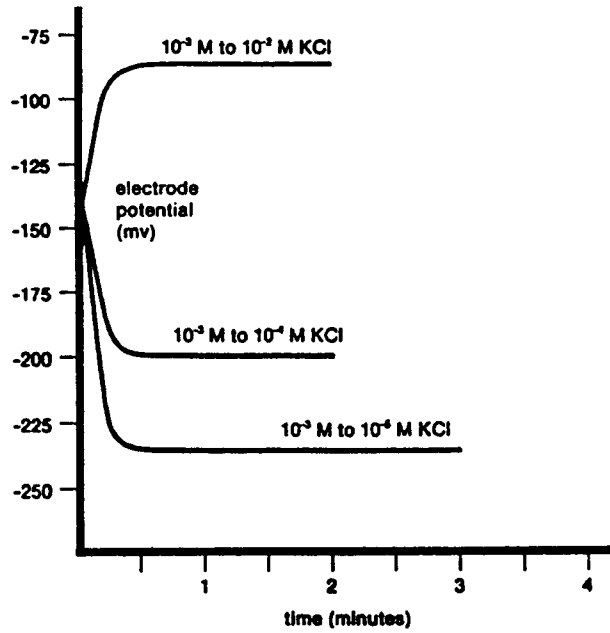


Figure 4-1 Typical Electrode Response to Step Changes in KCl

SECTION 5 TROUBLESHOOTING

SYMPTOM	POSSIBLE CAUSES	NEXT STEP
Out of range reading	Defective meter	Check meter with shorting strap
	Defective sensing module, electrodes not plugged in properly	Check electrode operation unplug electrodes and reseal
	Reference electrode not filled	Be sure reference electrode is filled
	Air bubble on membrane	Remove bubble by redipping electrode
	Calibration control not turned far enough	Continue turning the calibration control
"Incorrect Answers" (but calibration curve is good)	Incorrect scaling of semilog paper	Plot millivolts on the linear axis. On the log axis, be sure concentration numbers within each decade are increasing with increasing concentration
	Incorrect sign	Be sure to note sign of millivolt number correctly
	Incorrect standards	Prepare fresh standards
	Wrong units used	Apply correct conversion factor: $10^{-3}M = 39.1 \text{ ppm } K^+ = 74.6 \text{ ppm as } KCl$
	Complexing agents in sample	Use known addition, titration, or decomplexing procedure

SYMPTOM	POSSIBLE CAUSES	NEXT STEP
Drift(reading slowly changing in one direction)	Samples and standards at different temperatures	Allow solutions to come to room temperature before measurement
	Electrode exposed to interferences	Soak electrode in potassium standard
	Incorrect reference filling solution	Use recommended filling solution
Low Slope or No Slope	Standards contaminated or incorrectly made	Prepare fresh standards
	ISA not used	Use recommended ISA
	Standard used as ISA	Use ISA
	Defective sensing module	Check electrode operation
	Electrode exposed to interferences	Soak electrode in potassium standard
Noisy or unstable readings (readings continuously or rapidly changing)	Defective meter	Check meter with shorting strap
	Air bubble on membrane	Remove bubble by redipping electrode
	Wrong reference electrode	Do not use calomel or Ag/AgCl (frit or or fiber-type reference electrode
	ISA not used	Use recommended ISA
	Meter or stirrer not grounded	Ground meter or stirrer

SECTION 6 SPECIFICATIONS

CONCENTRATION RANGE: 1M to 1×10^{-6} M (3.9×10^4 ppm to 4.0×10^{-2} ppm)

pH RANGE: 2 to 12 (See sections on pH Effects and Electrode Operation)

TEMPERATURE RANGE: 0° to 40°C (continuous)
40° to 50°C (intermittent)

RESISTANCE: 10 -20 Mohm

REPRODUCIBILITY: +/-2%

SAMPLES: Aqueous solutions only
110 organic solvents

SIZE: 110 mm in Length
12 mm in Diameter
1 m cable Length)

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HEATERS

- ☑ Heating Cable
- ☑ Cartridge & Strip Heaters
- ☑ Immersion & Band Heaters
- ☑ Flexible Heaters
- ☑ Laboratory Heaters