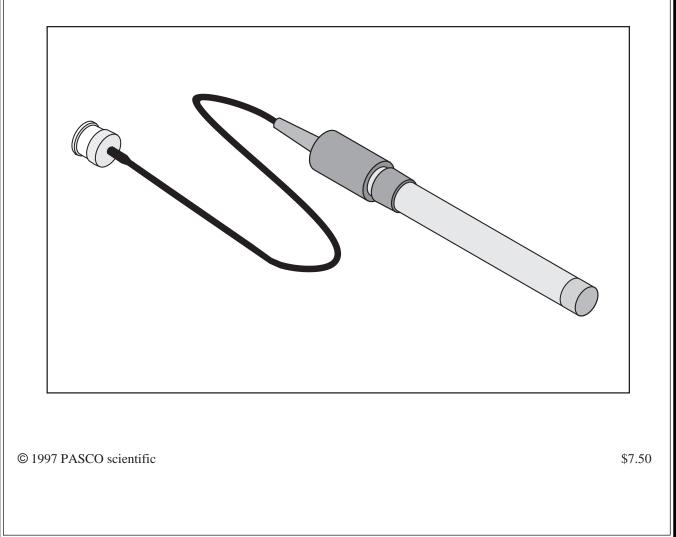
Instruction Manual and Experiment Guide for the PASCO scientific Model CI-6733

> POTASSIUM ION SELECTIVE ELECTRODE





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Always use eye protection and gloves when working with chemicals.



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- ③ Make certain that the packing material cannot shift in the box or become compressed, allowing the instrument come in contact with the packing carton.

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Introduction

The PASCO Potassium Ion Selective Electrode is used to quickly, simply, accurately, and economically measure Potassium ion concentration in aqueous solutions.

Theory

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 ion selective exchanger.

An electrode potential develops across the membrane when the membrane is in contact with a potassium solution. This electrode potential measured against a constant reference potential, using an ISE Amplifier and *Science Workshop* interface, 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:

where:

 $E = E_0 + S \log X$

E = measured electrode potential

 E_0 = reference potential (a constant)

S = electrode slope (\sim 56 mV/decade)

X = activity of potassium ions in solution

The activity, X, represents the effective concentration of free potassium ion in the solution. Both bound, C_b , and free, C_f , potassium ions are included in the total potassium ion concentration, C_t . The potassium ion electrode will only respond to free potassium ions, the concentration of which is:

$$C_f = C_t - C_b$$

The activity is related to the free lead ion concentration, C_{ρ} , by the activity coefficient, γ , by:

 $X = \gamma C_{f}$ Activity coefficients vary, depending on total ionic strength, I, defined as:

> $I = \frac{1}{2} \sum C_x Z_x^2$ where: $C_x = \text{concentration of ion } X$ $Z_x = \text{ charge of ion } X$ $\sum = \text{ sum of all of the types of ions in the solution}$

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



Strongly acidic (pH=0-2) and strongly basic (pH=12-14) solutions are also 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 sample or use a known increment method for ion measurement.

Equipment

Included:

- Potassium Ion Selective Electrode
- Potassium Ion Selective Electrode fill solution
- · pipette for fill solution

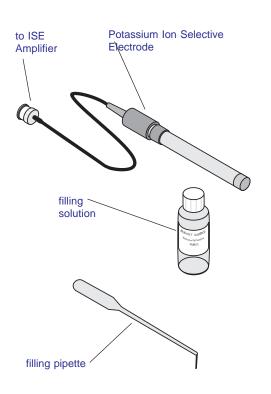


Figure 1. Included Equipment

Additional Required:

Required Equipment

- PASCO CI-6738 ISE (Ion Selective Electrode) Amplifier
- Science Workshop 2.2.5 or higher
- PASCO Science Workshop Computer Interface
- Semi-logarithmic 4-cycle graph paper for preparing calibration curves (Linear graph paper is recommended for low level measurements of potassium titrations.)
- magnetic stir plate

Required Solutions

The stock solutions listed in this section may be created as described in the text or ordered directly from PASCO. The solutions available for order and their respective prices are listed on the 'ISE Working Solutions Price List'.

- Deionized or distilled water for solution and standard preparation.
- Ionic Strength Adjuster (ISA), 5 M NaCl

To prepare this solution, half fill a one liter volumetric flask with distilled water and add 29.2 grams of reagent-grade sodium chloride (NaCl·H₂O). Swirl the flask gently to dissolve the solid. Fill the flask to the mark with distilled water, cap, and upend several times to mix the solution. To each 100 ml of standard or sample, add 2 ml of ISA. The background ionic strength of the resulting solution will be 0.1 M. This solution is used to adjust the pH and keep a constant background ionic strength present in the solution.

• Potassium Standard, 0.1M KCl

To prepare this solution, half fill a one liter volumetric flask with distilled water and add 7.46 grams of reagent-grade potassium chloride. Swirl the flask gently to dissolve the solid. Fill the flask to the mark with distilled water, cap, and upend several times to thoroughly mix the solution.



Potassium Standard, 1000 ppm

To prepare this solution, half fill a one liter volumetric flask with distilled water and add 256 ml of the 0.1 M standard. Swirl the flask to dissolve the solid. Fill the flask to the mark with distilled water, cap, and upend several times to thoroughly mix the solution standard.

General Preparation

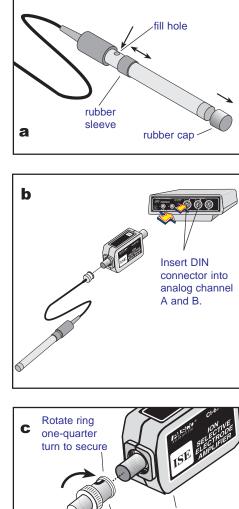
Electrode Preparation

- *1.* Remove the rubber cap covering the electrode tip. Slide the rubber sleeve down away from the filling hole of the Potassium Ion Selective Electrode. Fill the electrode with the included filling solution to a level just below the fill hole. Slide the rubber sleeve back over the filling hole (Figure 2a).
- 2. Connect the Potassium Ion Selective Electrode to the ISE Amplifier and insert the DIN connector of the ISE Amplifier into analog channel A or B on a PASCO Computer Interface (Figures 2b and 2c).

Electrode Slope Check Using ScienceWorkshop (check electrodes each day)

- 1. 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. Start Science Workshop, select the Ion Selective Electrode sensor, open a Digital display, and begin monitoring data. Lower the electrode tip into the solution.
- 2. Using a pipette, add 1 ml of 0.1 M or 1000 ppm potassium standard to the beaker. When the reading has stabilized, record the voltage reading indicated in the Digits display.
- 3. Using a pipette, add 10 ml of the same potassium standard used above to changed by $56 \pm 2 \,\mathrm{mV}$, assuming the temperature is between 20 °C and 25 °C. See the Troubleshooting sections if the potential change is not within this range.

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



ISE Amplifier electrode connector

Figure 2

Equipment Setup. a: filling the electrode with filling solution; **b & c**: connecting the electrode to the ISE Amplifier and to the computer interface



Measurement

Measuring Hints

- All samples and standards should be at the same temperature for precise measurement, preferably ambient temperature. A slight change in temperature may result in a substantial measurement error.
- 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 insulation material, such as a styrofoam sheet, between the stirrer and beaker.
- Always rinse the electrodes with distilled water and blot dry between measurements. Use a clean, dry tissue to prevent cross-contamination.
- A slow or sluggish electrode response may indicate surface contamination of the potassium electrode 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.
- For samples with high ionic strength, prepare standards whose composition is similar to the sample. Dilute concentrated samples (>0.1 M) before measurement.
- Use fresh standards for calibration.
- Use 2 ml of ISA for each 100 ml of sample or standard.
- Always check to see that the membrane is free from air bubbles after immersion into the standard or sample. Agitate the electrode gently to remove the air bubbles.

Sample Requirements

- All samples must be aqueous and not contain organics which can dissolve the epoxy electrode body and/ or the cement bonding the sensing crystal to the electrode body.
- The temperature of the standard solutions and of the sample solutions should be the same and below 40 °C with intermittent measurements allowed to 50°C. About a 2% error in the slope will occur for each 1 °C difference in temperature.
- Interferences found in table 3 should be absent. If they are present, use the procedure found in the *Interferences* and *Electrode Response* sections to remove them.

Units of Measurement

Potassium concentrations are measured in units of ppm as potassium, moles per liter, or any other convenient concentration unit. Table 1 indicates some concentration units and conversion factors.

TABLE 1 Concentration Unit Conversion Factors			
	ррт К ⁺¹	ppm KCl	moles/liter K ⁺¹
	3.91	7.46	1.0 X 10 ⁻⁴
	39.1	74.6	1.0 X 10 ⁻³
	391	746	1.0 X 10 ⁻²



Measurement Procedure

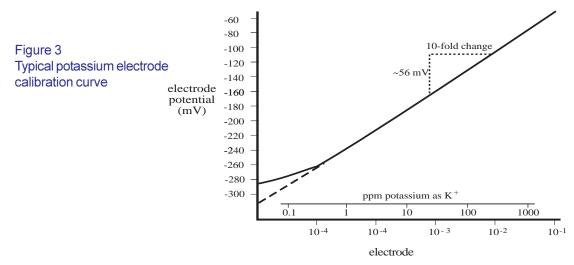
Direct Measurement

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 approximately the same by adjustment with ISA. The temperature of both sample solution and standard solution should be the same.

Direct Measurement of Potassium

► Note: A calibration curve is constructed on semi-logarithmic paper. The measured electrode potential (linear axis) is plotted against the standard concentration (log axis). In the <u>linear region of the curve, only two standards are necessary</u> to determine a calibration curve. Calibration standards close to the anticipated value of the "unknown" should be chosen. In the <u>non-linear region, additional points must be measured</u>. 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.

- *1.* By serial dilution, prepare 10⁻² M, 10⁻³ M, and 10⁻⁴ M or 100 ppm, 10 ppm and 1 ppm standards, from the 0.1 M or 1000 ppm standards. Prepare standards with a composition similar to the samples if the samples have an ionic strength above 0.1 M, add 2ml of ISA per 100 ml of standard.
- 2. Place the most dilute solution (10⁻⁴ M or 1 ppm) on the magnetic stirrer and begin stirring at a constant rate. After assuring that *Science Workshop* is operating, lower the electrode tip into the solution. When the reading has stabilized, record the voltage reading indicated in the Digits display.
- 3. Place the midrange solution $(10^{-3}$ M or 10 ppm) on the magnetic stirrer and begin stirring. After rinsing the electrodes with distilled water, blot dry, and immerse the electrode tip in the solution. When the reading has stabilized, record the voltage reading indicated in the Digits display.
- 4. Place the most concentrated solution (10⁻²M or 100 ppm) on the magnetic stirrer and begin stirring. Add 2 ml of ISA. After rinsing the electrodes with distilled water, blot dry, and immerse the electrode tip in the solution. When the reading has stabilized, record the voltage reading indicated in the Digits display.
- 5. Using the semi-logarithmic graph paper, plot the voltage reading (linear axis) against the concentration (log axis). Extrapolate the calibration curve down to about 2.0×10^{-6} M. A typical calibration curve can be found in Figure 3.



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. Rinse the electrode with distilled water electrode tip in the solution. Rinse the electrodes with distilled water, blot dry, and immerse the electrode tip in the solution. When the reading has stabilized, record the voltage reading indicated in the Digits display.
- 7. The electrode should be re-calibrated every 1-2 hours. Simply repeat steps 2-5 above.

Low Level Potassium Determination

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

- 1. Using 20 ml of standard ISA, dilute to 100 ml with distilled water.
- 2. Dilute 1 ml of 0.1 M standard to 100 ml to prepare a 1.0 X 10⁻³ M standard solution for measurements in moles per liter. Prepare a 100 ppm standard solution by diluting 10 ml of the 1000 ppm standard for measurements in ppm. Standards should be prepared fresh daily.
- *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 Science Workshop is operating.
- 5. Add increments of the $1.0 \times 10^{-3} \text{ M}$ or 100 ppm standard as given in Table 2 below.
- 6. After the reading has stabilized, record the voltage reading indicated in the Digits display.

TABLE 2: Step-wise Calibration For Low Level Potassium Measurements

		Added	Concentr	ation	
Step	Pipette	Volume (ml)	Μ	ppm	
1	А	0.1	1.0 X 10 ⁻⁶	0.1	
2	А	0.1	2.0 X 10 ⁻⁶	0.2	
3	А	0.2	4.0 X 10 ⁻⁶	0.4	
4	А	0.2	6.0 X 10 ⁻⁶	0.6	
5	А	0.4	9.9 X 10 ⁻⁶	1.0	
6	В	2.0	2.9 X 10 ⁻⁵	2.9	
7	В	2.0	4.5 X 10 ⁻⁵	4.8	

Pipet A = 1 ml graduated pipette

Pipet B = 2 ml pipette

Solutions: additions of 100 ppm or 1.0 X 10⁻³ M standard to 100 ml of distilled water and 1 ml of low level ISA.

7. On semi-logarithmic graph paper, plot the voltage reading on the Digits display (linear axis) against the concentration (log axis) as in Figure 3.



- 8. Rinse the electrodes in distilled water and blot dry.
- 9. Measure out 100 ml of the sample into a 150 ml plastic beaker and add 1 ml low level ISA. Place the beaker on the magnetic stirrer and begin stirring at a constant rate. Lower the electrode tip into the solution. After the reading has stabilized, record the voltage reading indicated in the Digits display and determine the concentration from the low level calibration curve. Prepare a new low level calibration curve daily. Check the calibration curve every 1-2 hours by repeating steps 3–7 above.

Electrode Characteristics

Reproducibility

Electrode measurements reproducible to $\pm 2\%$ 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.

Interferences

Table 3 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(s) in distilled water for an hour, then for two hours in potassium standard solution to restore proper response.

TABLE 3:Concentration of Possible Interferences Causing a 10% Error at VariousLevels of KCI; Background Ionic Strength is 0.12M NaCI.

Interferences			
(moles/liter)	10 ⁻² M K ⁺¹	10 ⁻³ M K ⁺¹	10 ⁻⁴ M K ⁺¹
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



interferences			
(ppm)	100 ppm K ⁺¹	10 ppm K ⁺¹	1 ppm K ⁺¹
Cs^{+1}	$1.0X10^{2}$	$1.0X10^{1}$	1.0
NH_4^{+1}	$2.7X10^{2}$	$2.7 X 10^{1}$	2.7
T1 ⁺¹	$3.14X10^{3}$	3.14×10^2	3.14X10 ¹
H ⁺¹	1.6pH	2.6pH	3.6pH
Ag ⁺¹	2.765X10 ⁵	$2.765 X 10^4$	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

Interferences

Temperature Influences

Samples and standards should be within $1 \,^{\circ}$ C of each other, since electrode potentials are influenced by changes in temperature. A 1 $^{\circ}$ C difference in temperature results in a 2% error at 1.0×10^{-3} M level. Because of the solubility equilibria on which the electrode depends, the absolute potential of the reference electrode changes slowly with temperature. The slope of the electrode, as indicated by the factor "S" in the Nernst equation, also varies with temperature. Table 3 gives values for the "S" factor in the Nernst equation for the lead ion. If changes in temperature occur, the electrodes should be recalibrated.

Provided that temperature equilibria has occurred, the potassium ion electrodes can be used at temperatures from 0^{0} C - 40^{0} C continuously and 40^{0} C - 50^{0} C intermittently. Room temperature measurements are recommended, since measurements at temperatures markedly different from room temperature may require equilibrium times up to one hour. Table 4 indicates the variation of theoretical slope with temperature.

TABLE 4: Temperature vs.Theoretical Values for theElectrode Slope

Temp (°C)	S (slope)
0	54.20
10	56.18
20	58.16
25	59.16
30	60.15
40	62.13
50	64.11

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 56 mV per decade. (Refer to Figure4)

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 10×10^{-5} M to several minutes near the detection limit.

Limits of Detection

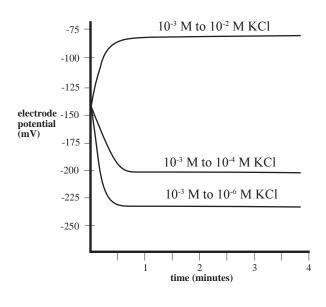
The upper limit of detection in pure potassium chloride solutions is 0.1 M. In the presence of other ions, the upper limit of detection is above 10 X 10^{-1} M of potassium. However, two factors influence this upper limit. Both the possibility of a liquid junction potential developing at the reference electrode and the salt extraction effect influence this upper limit. Some salts may extract into the electrode membrane at high salt concentrations, causing deviation from the theoretical response. Either dilute samples between 1 M and 1.0×10^{-1} M or calibrate the electrode at 4 or 5 intermediate points.



Figure 4

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Typical electrode time response to step changes in KCI
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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. A low level measurement is recommended if potassium measurements are made below 1.0 X 10^{-5} M (0.39 ppm as potassium).



pH Effects

Hydrogen ion interferes with low levels of potassium ion although the electrode can be used over a wide pH range. Table 3 should be used to determine the minimum pH at which low level potassium measurements can be made without more than a 10% error due to hydrogen ion interference.

Maintenance

Electrode Storage

The Potassium Ion Electrode may be stored in 1.0×10^{-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.

Specifications

Concentration Range:	1M to $1X10^{-6}$ M (3.9X10 ⁴ ppm to $4.0X10^{-2}$ ppm)
pH Range:	2 to 12 (See sections on pH Effects and Electrode Operation)
Temperature Range:	0^{0} to 40^{0} C (continuous) 40^{0} to 50^{0} C (intermittent)
Resistance:	10 -20 M ohm
Reproducibility:	+2%
Samples:	aqueous solutions only; no organic solvents
Size:	110 mm length12 mm diameter1 m Cable length



Troubleshooting Guide

The goal of troubleshooting is the isolation of a problem through checking each of the system components in turn: the electrodes, the standards and reagents, the sample, and the technique.

Electrode

The electrodes may be checked by using the procedure found in the sections entitled *Electrode Slope Check*.

- *1.* Be sure to use distilled or deionized water when following the procedures given in *Electrode Slope Check*.
- If the electrode fails to respond as expected, see the sections *Measuring Hints* and *Electrode Response*. Repeat the slope check.
- 3. If the electrode still fails to respond as expected, substitute another potassium ion electrode that is known to be in good working order for the questionable electrode.
- 4. If the problem persists, the reagent may be of poor quality, interferences in the sample may be present or the technique may be faulty.
- 5. If another electrode is not available for test purposes, or if the electrode in use is suspect, review the instruction manual and be sure to:
 - Clean and rinse the electrodes thoroughly.
 - Prepare the electrodes properly.
 - Use the proper filling solution.
 - Adjust the pH and the ionic strength of the solution by the use of the proper ISA.
 - Measure correctly and accurately.
 - Review Troubleshooting Hints.

Standards & Reagents

Whenever problems arise with the measuring procedure that has been used successfully in the past, be sure to check the reagent solutions. If in doubt about the credibility of any of the reagents, prepare them again. Errors may result from contamination of the ISA, incorrect dilution of the standards, poor quality distilled/deionized water, or a simple mathematical miscalculation.

Sample

Look for possible interferences, complexing agents, or substances which could affect the response or physically damage the sensing electrode if the electrodes work perfectly in the standard, but not in the sample.

Try to determine the composition of the samples prior to testing to eliminate a problem before it starts. (See *Measuring Hints*, *Sample Requirements*, and *Interferences*.)

Technique

Be sure that the electrode's limit of detection has not been exceeded. Be sure that the analysis method is clearly understood and is compatible with the sample.

Refer to the instruction manual again, particularly the *General Preparation* and *Electrode Characteristics* sections. If trouble still persists, call PASCO Technical Support.



Troubleshooting Hints

Symptom	Possible Causes	Next Step
Out of Range Reading	defective electrode	check electrode operation
	not plugged in properly	unplug electrode and reseat electrode
	reference electrode not filled	be sure reference electrode is filled
	air bubbles on membrane	remove bubble by redipping electrode
Noisy or Unstable	air bubble on membrane	remove bubble by redipping electrode
Readings (readings continuously or rapidly changing)	electrode exposed to interferences	soak electrode in potassium standard
	defective electrode	replace electrode
	ISA not used	use recommended ISA
	stirrer not grounded	ground stirrer
Drift (reading slowly changing in one direction)	samples end standards at different temperatures	allow solution 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
	standard used as ISA	use ISA
	ISA not used	use recommended ISA
	electrode exposed to interferences	soak electrode in potassium standard
	air bubble on membrane	remove bubble by redipping probe
"Incorrect Answer" (but calibration curve is good)	incorrect scaling of semi-log paper	plot voltage potential 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 reading correctly
	incorrect standards	prepare fresh standards
	wrong units used	apply correct conversion factor: $10^{-3} M = 39.1 \text{ ppm } \text{K}^{+1} = 74.6 \text{ ppm } \text{KCl}$





Technical Support

Feedback

If you have any comments about the product or manual, please let us know. If you have any suggestions on alternate experiments or find a problem in the manual, please tell us. PASCO appreciates any customer feedback. Your input helps us evaluate and improve our product.

To Reach PASCO

For technical support, call us at 1-800-772-8700 (toll-free within the U.S.) or (916) 786-3800.

fax:	(916) 786-3292

e-mail: techsupp@pasco.com

web: www.pasco.com

Contacting Technical Support

Before you call the PASCO Technical Support staff, it would be helpful to prepare the following information:

➤ If your problem is with the PASCO apparatus, note:

- Title and model number (usually listed on the label);
- Approximate age of apparatus;
- A detailed description of the problem/ sequence of events (in case you can't call PASCO right away, you won't lose valuable data);
- If possible, have the apparatus within reach when calling to facilitate description of individual parts.

If your problem relates to the instruction manual, note:

- Part number and revision (listed by month and year on the front cover);
- Have the manual at hand to discuss your questions.

