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IDENTIFIERS pH Calculation; *Waste Water Treatment; *Water Treatment

ABSTRACT

This document is an instructional module package prepared in objective form for use by an instructor familiar with pH, measurement of pH with a pH meter and maintenance of pH meter electrodes. Included are objectives, instructor guides, student handouts and transparency masters. This module considers the definition of pH, types of electrodes and their components, electrode maintenance, and procedures for using a pH meter to determine the pH of a water or wastewater sample. (Author/RH)

pH

Training Module 5.305.2.77

Prepared for the

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by

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September, 1977

The mention of trade names, or use of manufacturers technical bulletins, diagrams depicting specific equipment, or the commercial product in this module is for illustration purposes, and does not constitute endorsement or recommendation for use by Kirkwood Community College nor by the Iowa Department of Environmental Quality.

Module No:	Module Title: pH
Approx. Time:	Topics: Definition of pH pH Measurement System pH Measurement Electrode Maintenance Buffers

Objectives:

Upon completion of this module the participant should be able to:

1. Determine the pH of a sample using a pH meter and electrode.

Instructional Aids:

Overheads
Handout

Instructional Approach:

Lecture
Lab

References:

1. Willard, Merrit, Dean - Instrumental Methods of Analysis, 5th Ed., Chap. 20 D. Van Nostrand Co.
2. Modern Chemical Technology, Volume 3, American Chemical Society.

Class Assignments:

Instructional Aids

Overheads

Typed overheads are an example of overhead layout and content. For classroom use the overhead should be constructed using colored, 1/4 inch dry transfer letters.

Other overheads may be copied directly.

Handouts

Handouts may be copied directly.

Lab supplies and apparatus

Supplies and apparatus should be supplied per handouts so that participants may work in groups of 2 or 3.

Module No:	Module Title: pH Measurement
	Submodule Title:
Approx. Time: 30 Min.	Topic: Definition of pH

Objectives:

When the participant completes this module, they should be able to:

1. Define pH
2. Indicate that pH is a mathematical expression for the actual hydrogen ion concentration in water.
3. Explain that water dissociates into hydrogen ions and hydroxide ions and that the concentration of those two ions in pure water is 10^{-7} molar.
4. Indicate that a change in 1 pH unit is equivalent to a 10-fold change in hydrogen ion concentration

Instructional Aids:

Overheads

Instructional Approach:

Lecture

References:

1. Willard, Merrit, Dean - Instrumental Methods of Analysis, 5th Ed., Chap. 20, D. Van Nostrand Co.
2. Modern Chemical Technology, Volume 3, American Chemical Society.

Class Assignments:

Module No:	Topic: Definition of pH
Instructor Notes:	**Instructor Outline:**
Overhead Page ______	1. Define pH
	2. Indicate that pH is a mathematical expression for the actual hydrogen ion concentration in water.
Overhead Page ______	3. Explain that water dissociates into hydrogen ions and that the concentration of those two ions in pure water is 10^{-7} molar.
Overhead Page ______	4. Indicate that a change in 1 pH unit is equivalent to a 10-fold change in hydrogen ion concentration.
	Review logs

$$pH = -LOG[H^+]$$

OR

$$pH = LOG\frac{1}{[H^+]}$$

$$H_2O \leftrightarrow H^+ + OH^-$$

$$[H^+] = 0.0000007$$

$$= 1 \times 10^{-7}$$

TRANSPARENCY

TABLE 22-3

RELATIONSHIP OF (H^+), (OH^-), pH AND pOH

	(H^+)	pH	(OH^-)	
	1×10^0	0	1×10^{-14}	
	10^{-1}	1	10^{-13}	0.1 M HCl
	10^{-2}	2	10^{-12}	
	10^{-3}	3	10^{-11}	
	10^{-4}	4	10^{-10}	
	10^{-5}	5	10^{-9}	
	10^{-6}	6	10^{-8}	
NEUTRAL	10^{-7}	7	10^{-7}	DISTILLED WATER
	10^{-8}	8	10^{-6}	
	10^{-9}	9	10^{-5}	
	10^{-10}	10	10^{-4}	
	10^{-11}	11	10^{-3}	
	10^{-12}	12	10^{-2}	
	10^{-13}	13	10^{-1}	0.1 M NaOH
	10^{-14}	14	10^0	

TRANSPARANCY

NUMBER	1	2	3	4	5	6	7	8	9	10
LOGARITHM	0	.30	.48	.60	.70	.78	.84	.90	.95	1.0

NUMBER	LOGARITHM
1000	3.0
100	2.0
10	1.0
1	0.0
0.1	-1
0.01	-2
0.001	-3
0.0001	-4

Module No:	Module Title: pH Measurement
	Submodule Title:
Approx. Time: 30 Min.	Topic: pH Measurement System

Objectives:

Upon completion of this module, the participant should be able to:

1. Identify the parts of a pH electrode, reference electrode, and combination electrode.
2. Identify the 4 parts of the pH measurement system and indicate the weak links in the system.
3. Identify the controls and connection points on a common pH meter.

Instructional Aids:

Overheads

Instructional Approach:

Lecture

References:

1. Willard, Merrit, Dean - Instrumental Methods of Analysis, 5th Ed., Chap. 20 D. Van Nostrand Co.
2. Modern Chemical Technology, Volume 3, American Chemical Society.

Class Assignments:

Module No:	Topic:

Instructor Notes:	Instructor Outline:
Overhead Page_____	1. a. Describe the glass pH electrode
Overhead Page_____	b. Indicate how the electrode functions.
Overhead Page_____	c. Identify the parts of a pH electrode.
Overhead Page_____	d. Identify the parts of a reference electrode.
Overhead Page_____	e. Identify the parts of a combination electrode.
	2. a. Identify the 4 parts of the pH measurement system.
	3. Identify the controls and connection points on a common pH meter.
	List types of plugs on pH electrodes and on reference electrodes.
	List the controls and use.
	4. Discuss expanded scalepH meters___

TRANSPARANCY

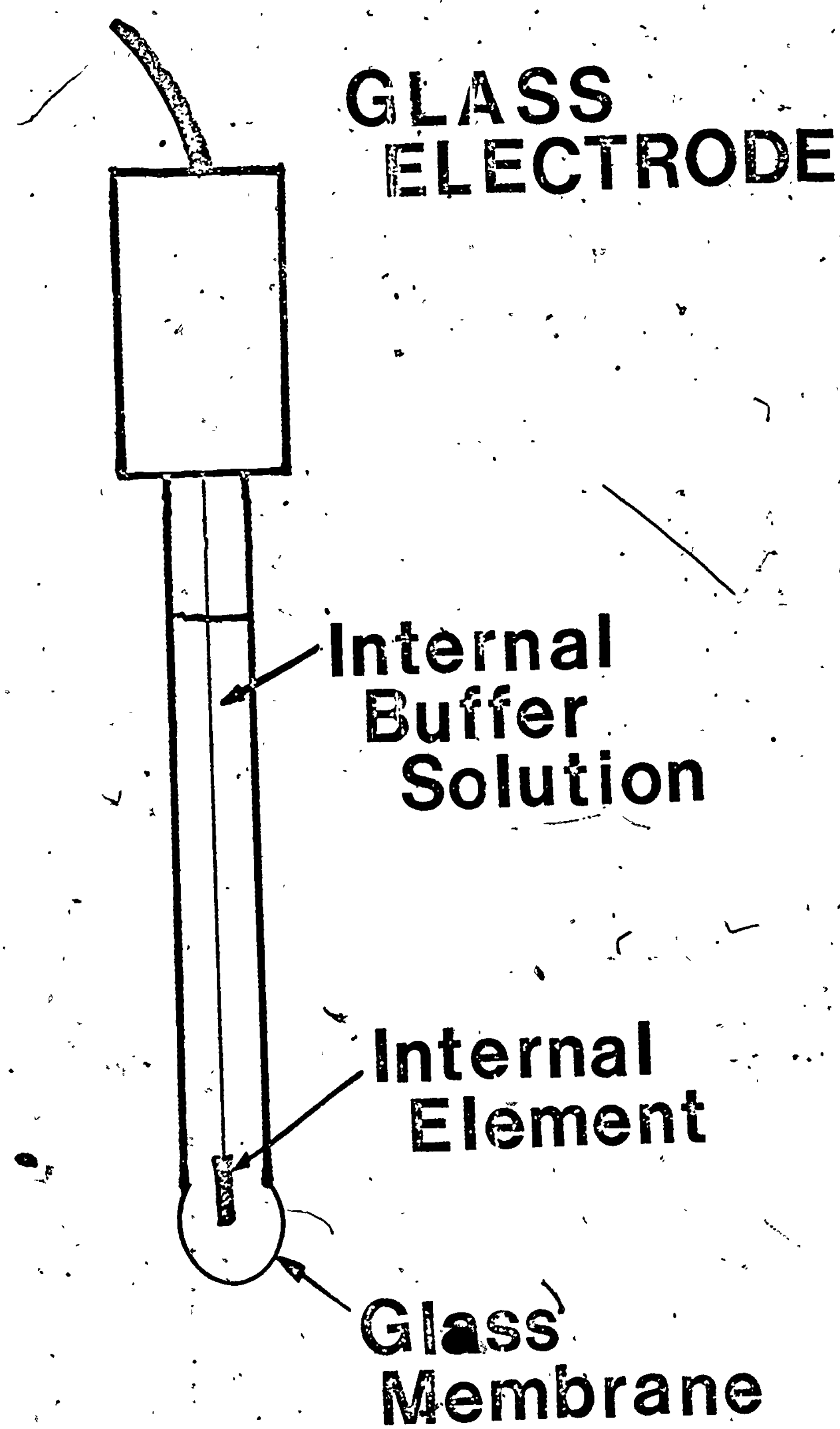
THE GLASS ELECTRODE COMPRISES A THIN-WALLED BULB OF CATION-RESPONSIVE GLASS SEALED TO A STEM OF NONCATION-RESPONSIVE, HIGH-RESISTANCE GLASS. THE ELECTRODE IS FILLED WITH AN ELECTROLYTE OF HIGH BUFFER CAPACITY AND INTO THIS BUFFER DIPS AN INNER REFERENCE ELECTRODE.

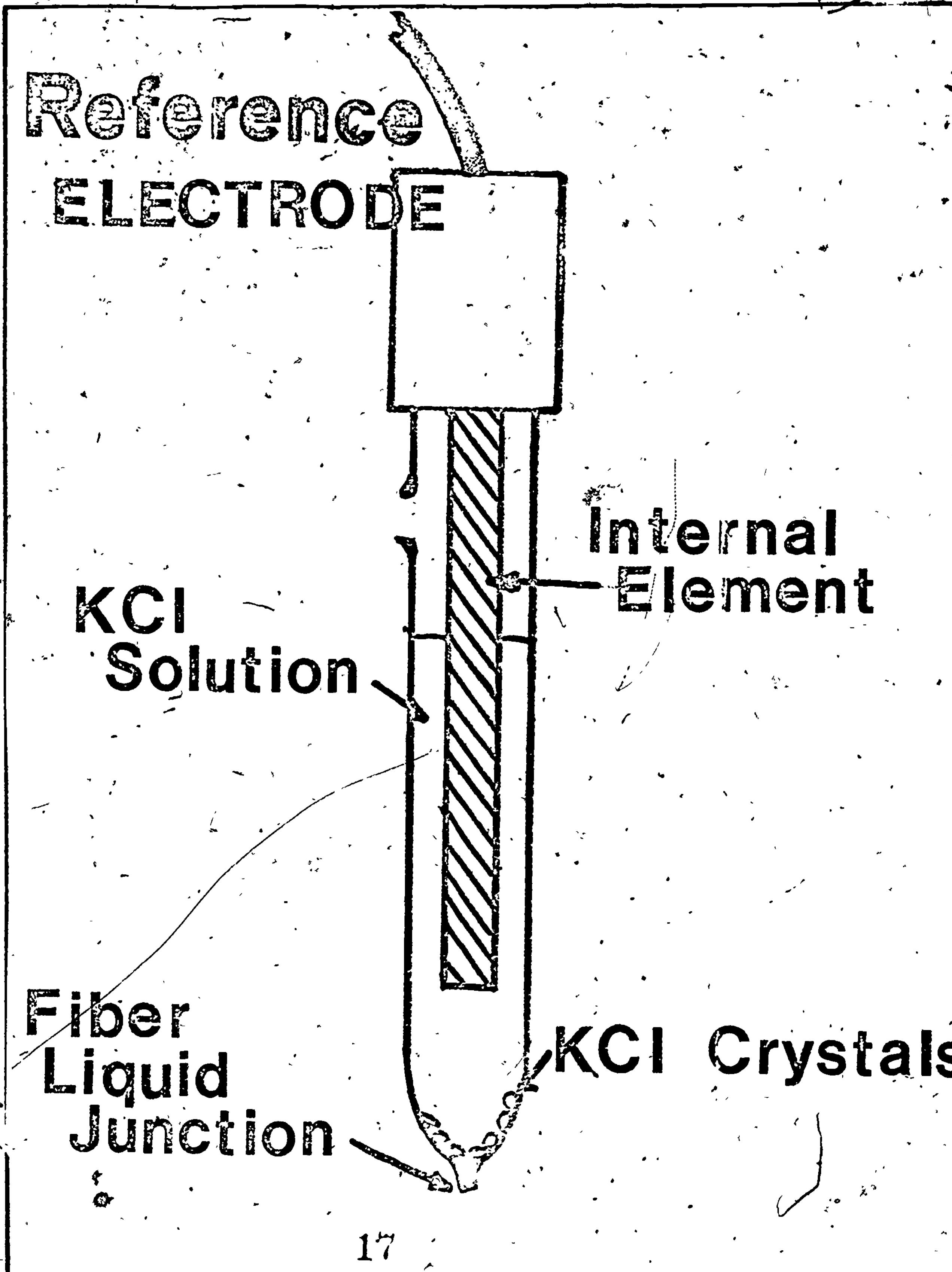
BOTH SURFACES OF THE GLASS MEMBRANE ARE CATION-RESPONSIVE. CHANGES IN THE ELECTRICAL POTENTIAL OF THE OUTER MEMBRANE SURFACE ARE MEASURED BY AN EXTERNAL REFERENCE ELECTRODE AND ITS ASSOCIATED SALT BRIDGE.

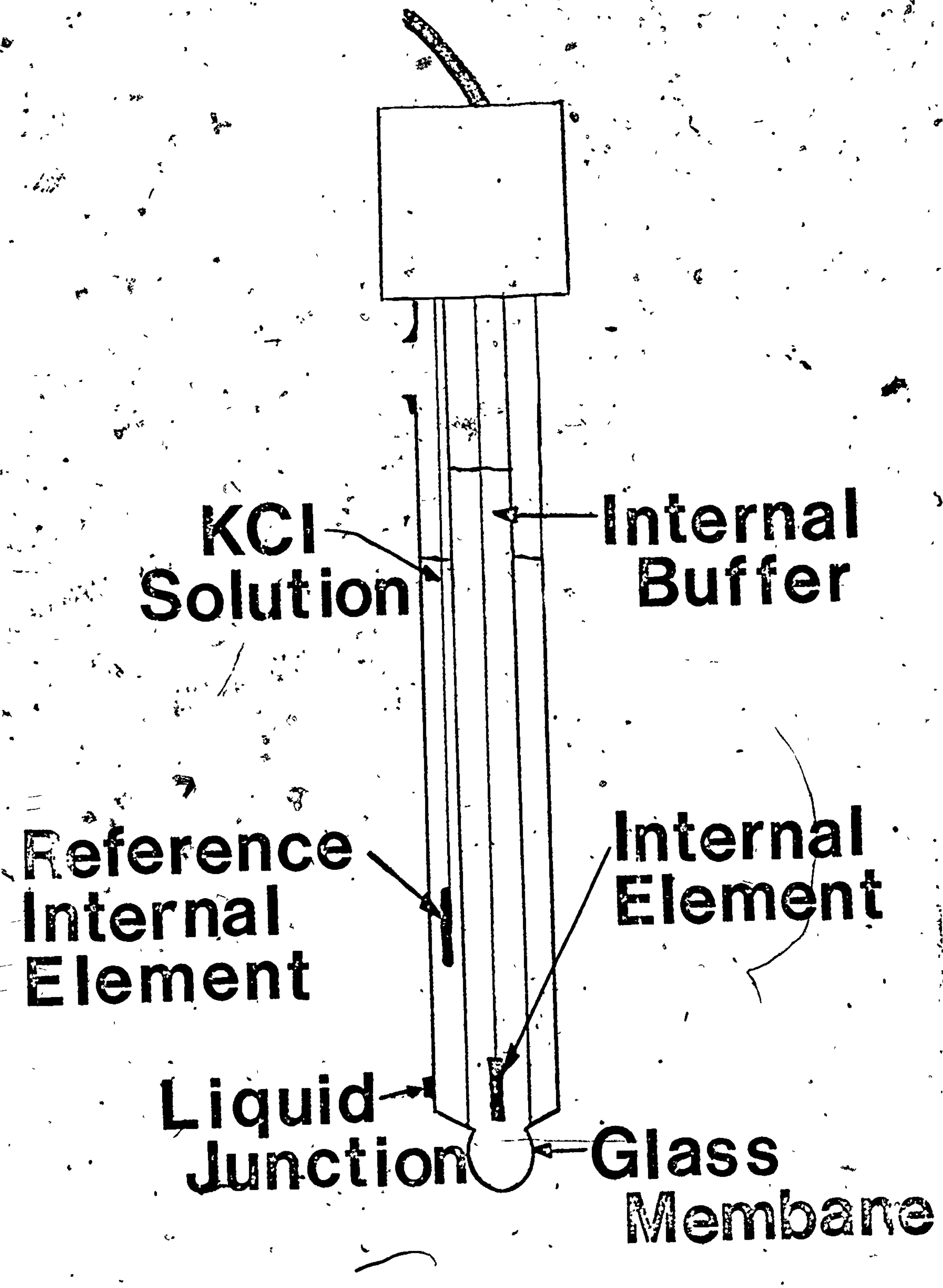
Internal Reference Electrode	Internal Buffer Solution	Glass Membrane	Standard or Unknown Solution	Salt Bridge	External Reference Electrode

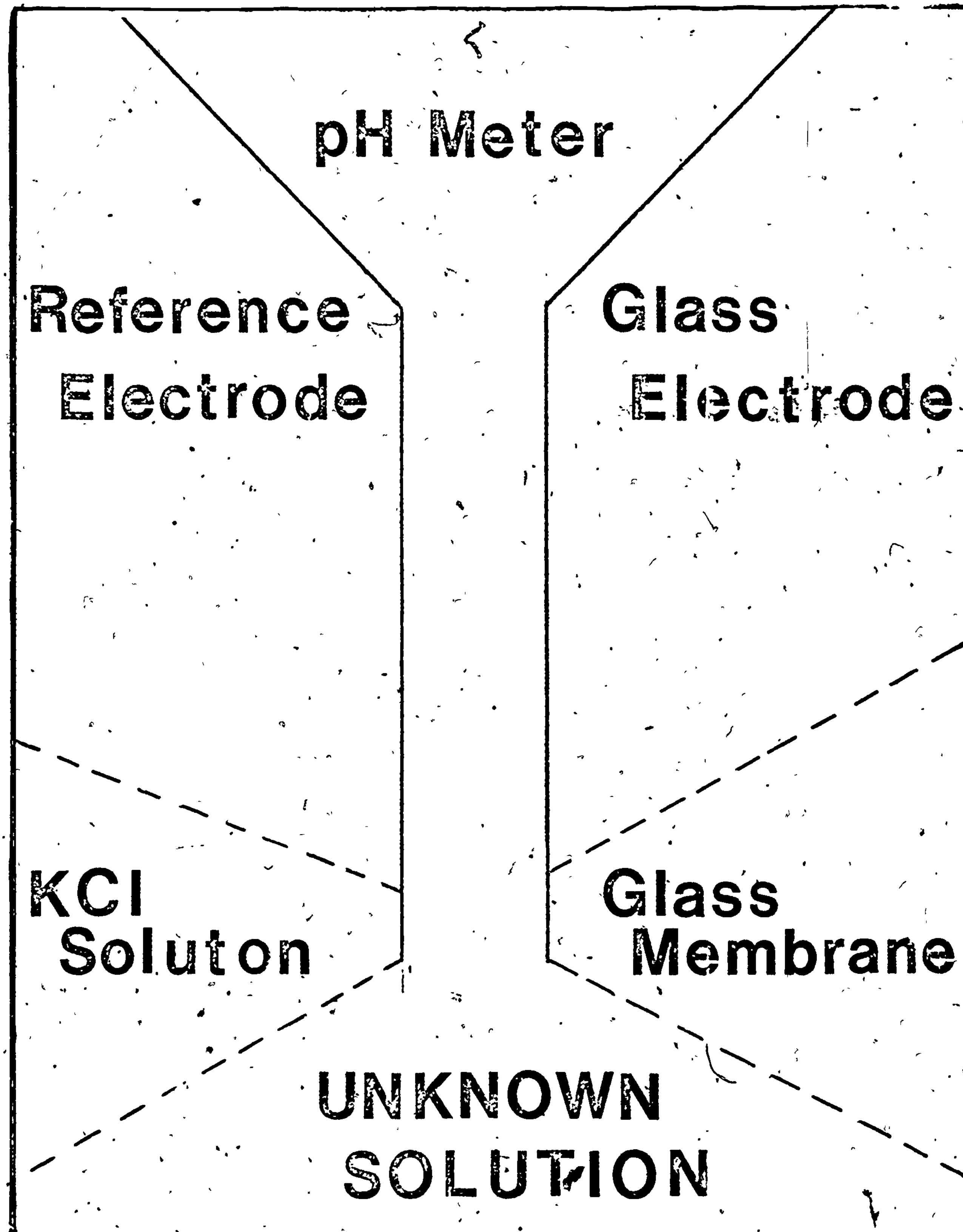
Internal Buffer Solution	Hydrated Glass-Gel Layer	Dry Glass Layer	Hydrated Glass-Gel Layer	Standard or Unknown Solution

TRANSPARENCY


GLASS ELECTRODE
Internal Buffer Solution
Internal Element
Glass Membrane


Reference
ELECTRODE
Internal
Element
KCl
Solution
Fiber
Liquid
Junction
KCl Crystals


KCl
Solution
Internal
Buffer
Reference
Internal
Element
Internal
Element
Liquid
Junction
Glass
Membane


pH Meter
Reference Electrode
Glass Electrode
KCl Soluton
Glass Membrane
UNKNOWN SOLUTION

pH METERS

CONTROLS AND CONNECTION POINTS

I. Off - stand-by - read switch

II. Calibration control

III. Temperatur control (slope)

IV. Glass electrode input

V. Reference electrode input

Optional

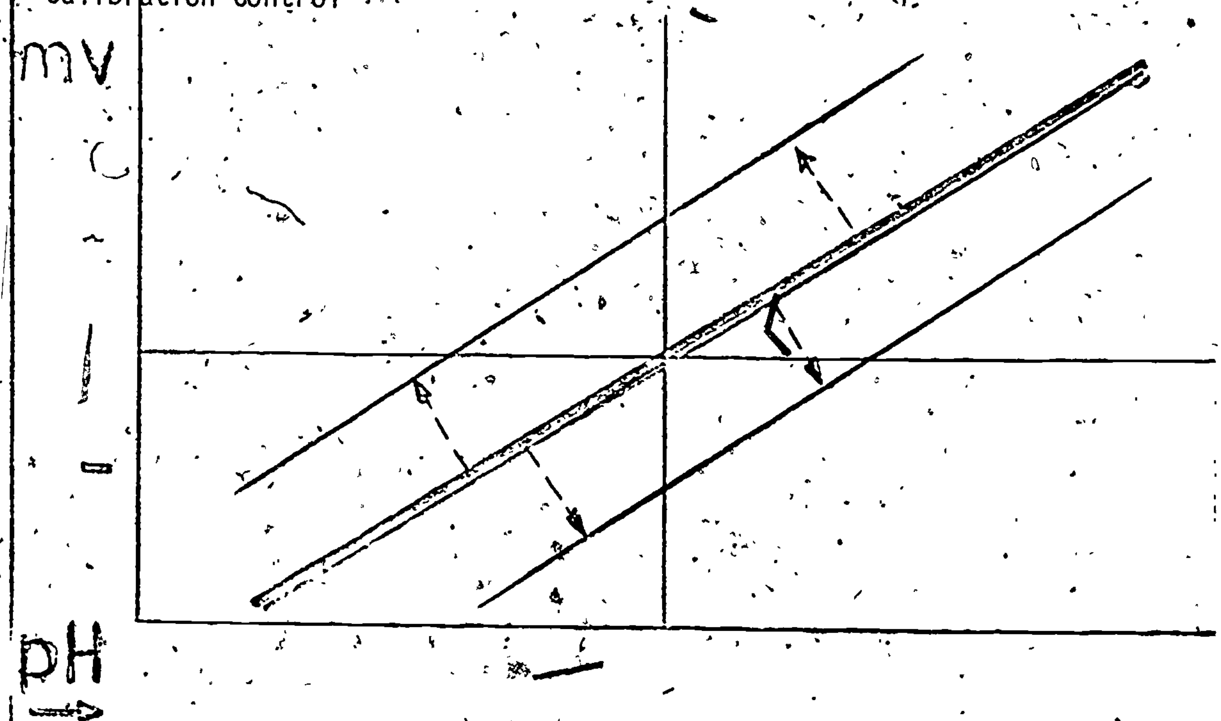
Milivolt switch

Auto-temperature control

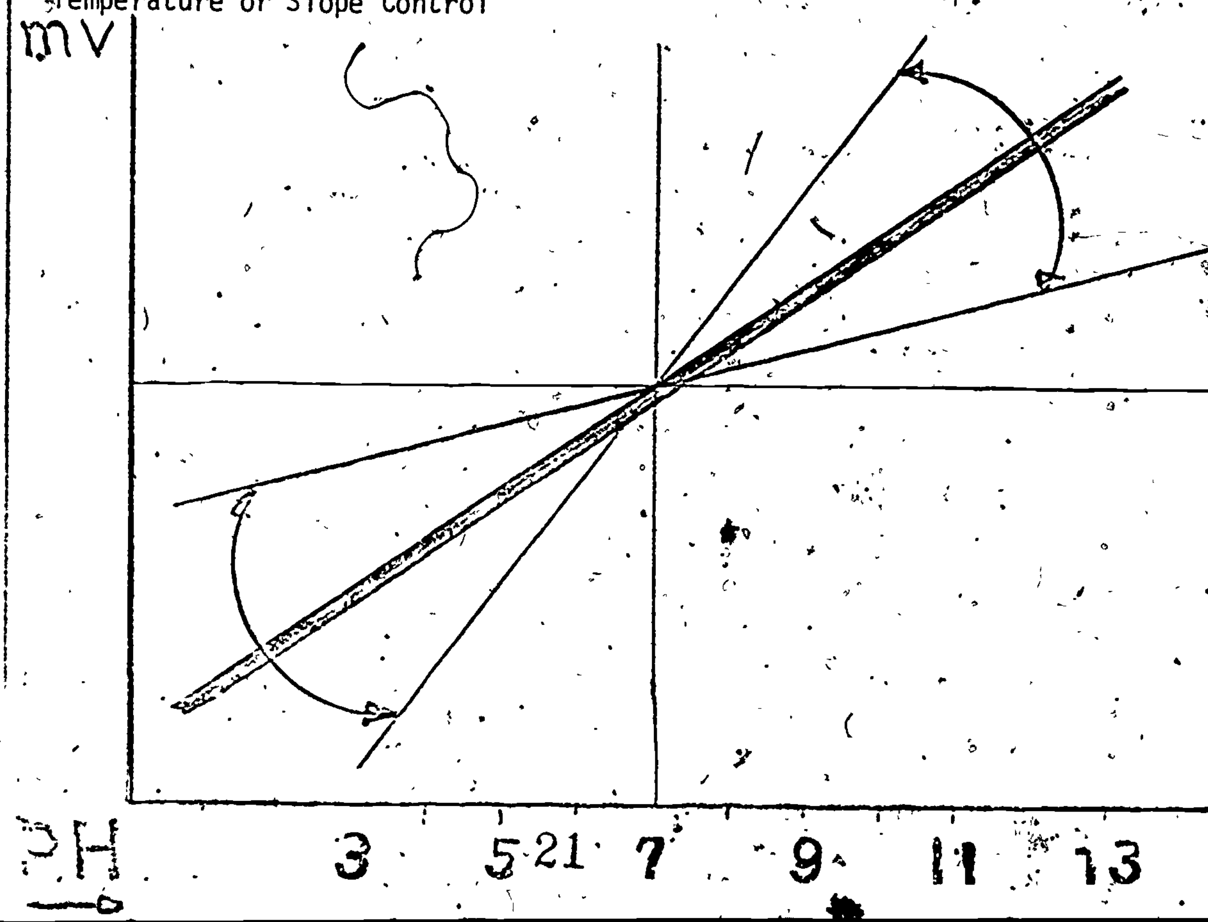
Expanded scale switch

Secondary slope control

Calibration Control



Temperature or Slope Control



TRANSPARANCY

FOR PRECISE WORK, EXPANDED SCALE PH METERS ARE AVAILABLE IN WHICH THE ENTIRE SCALE COVERS ONLY 1 OR 2 PH UNITS. ON THESE METERS THE PH CAN BE READ TO 0.005.

CAUTION: JUST BECAUSE A METER CAN BE READ TO 0.005 UNITS DOES NOT MEAN IT IS ACCURATE TO 0.005 UNITS. THE METER IS ONLY AS ACCURATE AS THE STANDARD USED TO CALIBRATE IT.

Module No:	Module Title: pH Measurement
	Submodule Title:
Approx. Time: 1 hour	Topic: pH Measurement

Objectives:

Upon completion of this module, the participant should be able to:

1. Standardize a pH meter using a single buffer.
2. Standardize a pH meter using a two buffered system.
3. Determine the pH of a highly buffered solution and an unbuffered solution.

Instructional Aids:

pH meter and electrode
pH buffers 4, 7, 9
Distilled waterer
Acetic acid 1% + Sodium acitate 1% solution

Instructional Approach:

Lab

References:

1. Willard, Merrit, Dean - Instrumental Methods of Analysis, 5th Ed., Chap. 20, D. Van Nostrand Co.
2. Modern Chemical Technology, Volume 3, American Chemical Society.

Class Assignments:

Module No:	Topic:

Instructor Notes:	Instructor Outline:
Overhead Page ___	1. Standardize a pH meter using a single buffer. 2. Standardize a pH meter using a two buffered system. 3. Determine the pH of a highly buffered solution and an unbuffered solution. 4. Have participants standardize a pH meter. 5. Have participants determine pH of some common solutions.

TRANSPARANCY

TO CALIBRATE THE METER, THE ELECTRODES ARE PLACED IN A SOLUTION OF KNOWN PH AND THE METER IS ADJUSTED TO READ THAT VALUE.

TO CHECK THE OPERATION OF THE METER AFTER CALIBRATION THE ELECTRODES ARE PLACED IN A SECOND BUFFER SOLUTION.

IF THE METER DOES NOT READ THE PH OF THE SECOND BUFFER SOLUTION, A SPECIAL FINE ADJUSTMENT CAN BE MADE. CHECK THE OPERATION MANUAL BEFORE YOU MAKE THIS ADJUSTMENT.

Module No:	Module Title: pH Measurement
	Submodule Title:
Approx. Time: 30 Min.	Topic: Electrode Maintenance

Objectives:

Upon completion of this module, the participant should be able to:

1. Clean and recondition a pH electrode.
2. Check the fiber junction of a reference electrode for flow.
3. Clean and recondition a reference electrode.

Instructional Aids:

Handout

Instructional Approach:

Lecture
Lab

References:

1. Willard, Merrit, Dean - Instrumental Methods of Analysis, 5th Ed., Chap. 20, D. Van Nostrand Co.
2. Modern Chemical Technology, Volume 3, American Chemical Society.

Class Assignments:

Module No:	Topic: Electrode
Instructor Notes:	Instructor Outline:
Handout Pages______	1. Clean and recondition a pH electrode. 2. Check the fiber junction of a reference electrode for flow. 3. Clean and recondition a reference electrode.

COMBINATION ELECTRODES

Principles

pH is a measure of the acidity of a solution and is defined as: $pH = -log_{10} + a_{H^+}$ is the activity of the hydrogen ion. The glass membrane of a pH electrode responds to the hydrogen ion activity by developing an electrical potential at the glass/liquid interface. At a constant temperature, this potential varies linearly with the pH of the solution being measured. The change in potential per pH unit is known as the slope of the electrode. This value increases linearly with temperature. Theoretical slope is known as the Nerstian slope and is identical to that developed by the hydrogen electrode. Slope value for pH electrodes approximate theoretical values very closely.

The combination electrode contains the pH half-cell, with a buffered salt solution sealed into the electrode body, and its own reference half-cell. The actual potential observed will be the sum of the separate potentials of the pH and reference half-cells. Since the potentials inside the pH electrode are fixed by the filling solution and the reference electrode potential is constant, any change in the potential of the electrode system at a given temperature will be due to changes in the pH of the solution being measured.

Temperature Effects

The effect of temperature on pH measurements depends on the reference electrode used, the pH of the solution within the pH electrode, and the pH of the test solution. At a certain pH, temperature will have little effect on the potential of an electrode system. This is known as the isopotential point. Also, at some pH, the system will exhibit no potential. This is known as the zero potential point. Both the isopotential and the zero potential point are features designed into the electrodes. Most electrodes are designed so that the isopotential and zero potential points are both near pH 7 to minimize the temperature effects at this calibration point.

General Information

All combination electrodes have Ag/AgCI internal elements for both the pH and the reference half-cells, and all have ceramic junctions.

Precautions

1. Prior to use, remove the protective cap and fill hole cover to allow the flow of electrolyte.
2. KCI crystals in either the electrode or in the filling solution container will not adversely affect either the potential or operation of the electrode. At room temperature, 4 M KCI is very close to saturation, and low ambient temperatures are sufficient to cause precipitation of some KCI crystals from the electrolyte solution. Excess KCI crystals may be removed by Procedure No. 1 Section 3.3. Brown particles in either the electrode or the filling solution are silver chloride. Their presence is an indication that the electrolyte solution is properly saturated with silver chloride.
3. If it is necessary to make electrolyte filling solution, high purity water should be used, along with reagent grade chemicals. AgCI is difficult to dissolve in 4 M KCI. Care should be taken to ensure that the solution

effect on the potential of an electrode system. This is known as the isopotential point. Also, at some pH, the system will exhibit no potential. This is known as the zero potential point. Both the isopotential and the zero potential point are features designed into the electrodes. Most electrodes are designed so that the isopotential and zero potential points are both near pH 7 to minimize the temperature effects at this calibration point.

General Information

All combination electrodes have Ag/AgCI internal elements for both the pH and the reference half-cells, and all have ceramic junctions.

Precautions

1. Prior to use, remove the protective cap and fill hole cover to allow the flow of electrolyte.
2. KCI crystals in either the electrode or in the filling solution container will not adversely affect either the potential or operation of the electrode. At room temperature, 4 M KCI is very close to saturation, and low ambient temperatures are sufficient to cause precipitation of some KCI crystals from the electrolyte solution. Excess KCI crystals may be removed by Procedure No. 1 under preventive maintenance section. Brown particles in either the electrode or the filling solution are silver chloride. Their presence is an indication that the electrolyte solution is properly saturated with silver chloride.
3. If it is necessary to make electrolyte filling solution, high purity water should be used, along with reagent grade chemicals. AgCI is difficult to dissolve in 4 M KCI. Care should be taken to ensure that the solution

is saturated, but not super-saturated, with AgCI.

4. When not is use, the protective cap that comes with the electrode should be filled with a dilute buffer and replaced for storage. The fill hole enclosure should also be replaced to prevent evaporation and slow the flow of electrolyte solution through the junction.
5. When transferring electrodes from one solution to another during measurements, rinse them with the solution to be measured next, or distilled water.
6. To optimize electrode performance, electrodes, buffers, and unknown samples should be equilibrated at the same temperature prior to measurements.
7. Electrical noise pick up may result if filling solution level is too low. Always maintain reference electrolyte above the fill hole.
8. For all side arm electrodes, if a high flow of electrolyte solution is desired or the electrode is to be used under external pressure conditions, the side arm enables the electrode to be internally pressurized. Pressurization may be accomplished by connecting a length of rubber tubing to the side-arm and the other end to a reservoir of electrolyte filling solution above the height of the side arm. Differential pressures of greater than 3 psi between the sample and the internal solution should be avoided.

Preventive Maintenance

1. The solution level should be maintained above the internal element at all times. If the solution is allowed to dry out, the excess salt crystals can be removed by rinsing out the electrode, first with hot distilled water, then rinse and refill with 4 M KCI saturated with AgCI. After this

treatment, the electrode should be allowed to soak in pH 7 buffer for several hours before being used again.

2. Under some circumstances, the ceramic reference junction may become clogged. This may result in unstable or drifting meter readings. The junction may be tested by taking a resistance reading (No. 3 below), or by wiping off the electrode tip and observing it after an hour of air drying. A high resistance or failure of saturated crystals to appear at the junction indicate a clogged junction. A clogged junction may be caused by AgCI precipitate in the junction. AgCI is highly insoluble in pure water and is best removed by soaking the electrode tip in hot saturated KCI solution. If the junction remains clogged, place it in a warm solution of dilute hydrochloric acid until it flows freely.
3. The resistance of the reference junction may be tested with an ohmmeter. Immerse the electrode into a beaker of saturated KCI. Connect one lead of the ohmmeter to the reference connector and the other lead of the ohmmeter to the reference connector and the other lead of the ohmmeter to the KCI solution. The resistance measured should be less than 50K.
4. If the pH bulb becomes contaminated or left dry, it may be reconditioned by placing the electrode tip in a 1.0 M solution of KOH for a few minutes, and then in a 1.0 M HCI for a few minutes. Rinse with distilled water, and soak in buffer solution.

Module No:	Module Title: pH Measurement
	Submodule Title:
Approx. Time: 30 Min.	Topic: Buffers

Objectives:

Upon completion of this module, the participant should be able to:

1. Differentiate between acidity, alkalinity and pH by discussion buffering and defining acidity, alkalinity, and pH.
2. Demonstrate schematically how a simple buffer system works.

Instructional Aids:

Overheads

Instructional Approach:

Lecture

References:

1. Willard, Merrit, Dean - Instrumental Methods of Analysis, 5th Ed., Chap. 20, D. Van Nostrand Co.
2. Modern Chemical Technology, Volume 3, American Chemical Society.

Class Assignments:

Module No:	Topic: Buffers

Instructor Notes:	Instructor Outline:
Overhead Pages ______ ______	1. Differentiate between acidity, alkalinity, and pH by discussion buffering and defining acidity, alkalinity and pH. 2. Demonstrate schematically how a simple buffer system works.

TRANSPARANCY

ALKALINITY - THE CAPACITY OF A SOLUTION TO NEUTRALIZE ACIDS. IT IS MEASURED BY TITRATION WITH STANDARD ACID TO A SPECIFIED pH.

ACIDITY - THE CAPACITY OF A SOLUTION TO NEUTRALIZE ALKALI. IT IS MEASURED BY TITRATION WITH STANDARD BASE TO A SPECIFIED pH.

IT IS EXPRESSED IN MILLIGRAMS PER LITER OF EQUIVALENT CALCIUM CARBONATE.

TRANSPARANCY

A BUFFER SOLUTION CONSISTS OF EITHER A WEAK ACID ALONG WITH A SALT OF THAT ACID OR A WEAK BASE PLUS A SALT OF THE BASE.

TRANSPARANCY

IN ORDER TO MAINTAIN A CONSTANT pH, A BUFFER IS USED. A BUFFER IS A SOLUTION OF A SUBSTANCE OR COMBINATION OF SUBSTANCES WHICH RESISTS A CHANGE IN pH EVEN WHEN A STRONG ACID OR BASE IS ADDED.

Module No:	Module Title:
	Submodule Title:
Approx. Time:	
	EVALUATION

Objectives:

Determine the pH of an unknown buffer solution given a pH meter electrode and standard buffers to an accuracy of ± .1 pH unit.