DCCUMENT RESUME

ED 153 868 SE 024 252

AUTHOR	Bonte, John L.; Davidscn, Arncló C.
TITLE	Alkalinity Analysis. Training Mcdule 5.22C.2.77.
SPONS AGENCY	Department cf Labor, Washingtcn, D.C.; Icwa State Dept. of Environmental Quality, Des Mcines.
PUB DATE	Sep 77
NOTE	33p.; For related documents, see SE 024 249-254; Contains marginal legibility in transparency masters
EDRS PRICE	MF-$0.83 HC-$2.C6 Plus Postage.
DESCRIPTORS	*Chemical Analysis; Chemistry; *Instructicnal Materials; *Labcratcry Procedures; *Laboratory Techniques; *Post Seccndary Educaticn; Secondary Education; Units of Study
IDENTIFIERS	*Alkalinity; *Water Treatment

ABSTRACT

This document is an instructicnal mcdule package prepared in objective fcrm fcr use by an instructcr familiar with the acid-base titrimetric procedure for determining the hydrcxide, carbonate and bicarbcnate alkalinity cf a water sample. Included are objectives, an instructor guide, student handouts and transparency masters. A video tape is also available frcm the authcr. This module considers use cf the pH meter, preparaticn and standardization cf reagents, titration and calculaticn cf results. (Authcr/RE)

ALKALINITY ANALYSIS

Training Module 5.220.2.77

Prepared for the

Iowa Department of Environmental Quality
Wallace State Office Building
Des Moines, Iowa 50319

by

John L. Bonte
Developer
Arnold C. Davidson
Project Director
Clinton Community College
1000 Lincoln Boulevard
Clinton, Iowa 52732

The publication of these training materials was financially aided through a contract between the Iowa Department of Environmental Quality and the Office of Planning and Programming, using funds available under the Comprehensive Employment and Training Act of 1973. However, the opinions expressed herein do not necessarily reflect the position or policy of the U. S. Department of Labor, and no official endorsement by the U. S. Department of Labor should be inferred.

September, 1977

Module No:	Module Title: Alkalinity Analysis
	Submodule Title:
Approx. Time: 3 hours	Topic: Summary

Instructional Objective:

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

1. Operate and standardize a pH meter.
2. Determine hydroxide, carbonate, and bicarbonate alkalinity of a water sample.
3. Prepare and standardize all reagents needed for an alkalinity analysis.

Instructional Aids:

Transparencies Ak1 - Ak6 - softening videotape

Instructional Approach:

Lecture, discussion, videotape viewing, laboratory practice.

References:

1. "Standard Methods for the examination of water and wastewater," 14th ed., pp. 278-282.
2. G.V.James, "Water Treatment," 3rd ed., London: Technical Press, 1965.

Class Assignments:

Module No:

Module Title:
Alkalinity Analysis

Submodule Title:

Approx. Time:
0.5 hours

Topic:
Definition of Alkalinity

Instructional Objective:

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

1. Write chemical reactions which explain alkalinity.
2. Expalin how alkalinity is used in water treatment.
3. Describe the difference between phenolophthalein and total alkalinity.
4. Describe the difference between hydroxide, bicarbonate, and carbonate alkalinity.
5. Relate pH to hydroxide and hydrogen ion concentrations.

Instructional Aids:

Transparency Ak1 - titration end points.

Transparency Ak2 - Relation of pH to concentration.

Softening video tape

Instructional Approach:

Lecture - discussion

References:

1. Standard Methods, p. 278
2. James, p. 107-122.

Class Assignments:

Module No: Ak	Topic: Definition of Alkalinity

Instructor Notes:	Instructor Outline:
Softening video tape	1. Alkalinity is the capacity of a water to neutralize a strong acid to a designated pH. a. Bicarbonate $HCO_3^- + H^+ \rightleftharpoons H_2CO_3$ b. Carbonate $CO_3^{2-} + 2H^+ \rightleftharpoons H_2CO_3$ c. Hydroxide $OH^- + H^+ \rightleftharpoons H_2O$
	2. Use of alkalinity a. alkalinity depends on: HCO_3^-, $CO_3^=$ and OH^- b. used to determine relative amount of lime and soda as to be used in softening c. used in anaerobic digesters
Transparency Ak-1 Titration end points	3. Phenol pthalein - titrate to 8.3 pH Total - titrate to lower pH
	4. Order of titration OH^-, CO_3^{2-}, HCO_3^-
Transparency Ak-2 Relation of pH to concentration	5. $pH = -\log(H^+)$ $pOH = 14 - pH$

Module No:	Module Title: Alkalinity Analysis
	Submodule Title:
Approx. Time: 0.25 hours	Topic: Use of the pH meters

Instructional Objective:

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

1. Describe the function of the pH meter.
2. Properly standardize the pH meter.
3. Determine the pH of a solution with a pH meter.

Instructional Aids:

Transparency Ak3 - diagram of pH meter.

Instructional Approach:

Lecture - demonstration

References:

Operator's manual for pH meter used.

Class Assignments:

Module No: Ak	Topic: Use of the pH meter
Instructor Notes:	**Instructor Outline:**
	1. pH meter measure the H^+ ion concentration of a solution electronically.
Transparency Ak-3 Diagram of pH meter	2. Standardize with pH 7 buffer solution to a meter reading of 7.0.
	3. Determine pH by placing electrodes in solution and reading meter.

Module No:	Module Title: Alkalinity Analysis
	Submodule Title:
Approx. Time: 0.25 Hours	Topic: Safety

Instructional Objective:

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

1. Locate the following in the laboratory and demonstrate proper use: emergency shower, fire extinguisher, eye wash, first aid kit.
2. Select and use safety glasses, lab coat or apron and gloves in the appropriate situation.
3. Describe the hazards associated with the chemicals used in the alkalinity determination.

Instructional Aids:

Hand-out of safety rules for the laboratory.

Instructional Approach:

Lecture/demonstration

References:

Basic laboratory skills module

Class Assignments:

Module No: Ak	Topic: Safety

Instructor Notes:	Instructor Outline:
	1. Point out to student all the safety equipment in the laboratory. 2. Point out the hazard of strong acids in the eye. 3. Electrical shock from pH meter and acid burns are the main hazards.

Module No:

Module Title:

Alkalinity Analysis

Submodule Title:

Approx. Time:

0.5 hours

Topic:

Preparation of reagents

Instructional Objective:

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

1. Prepare a 0.05 standard sodium carbonate solution and calculate its exact normality.
2. Prepare 0.1N and 0.02N hydrochloric acid solutions.

Instructional Aids:

Instructional Approach:

Laboratory practice

References:

Standard Methods, p. 279

Class Assignments:

Module No: Ak	Topic: Preparation of reagents
Instructor Notes:	**Instructor Outline:**
1. 2.5g per liter	1. Na_2CO_3 solution: N. =0.05 N $N = \frac{g\ Na_2CO_3\ per\ liter}{53}$
2.a. 0.1 N HCl 8.3 ml conHCl per liter	2. Acid a. o.1N hydrochloric acid
b. 0.02 N HCl 200 ml 0.1 NHCl per liter of solution	b. 0.02 N hydrochloric acid

Module No:

Module Title:

Alkalinity Analysis

Submodule Title:

Approx. Time:

0.5 hours

Topic:

Standardization of Acid

Instructional Objective:

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

1. Properly titrate the standard Na_2CO_3 solution with 0.1N HCl potentiometrically and construct a titration curve.
2. Identify inflection points.
3. Calculate from the titration curve the exact normality of the 0.1N HCl and the 0.02N HCl.
4. Calculate the $CaCO_3$ equivalence of the HCl solutions.

Instructional Aids:

Transparency Ak4 - sample Na_2CO_3 titration curve.

Instructional Approach:

Laboratory practice

References:

Standard Methods p. 279

Class Assignments:

Module No: Ak	Topic: Standardization of acid
Instructor Notes:	**Instructor Outline:**
Transparency Ak-4 Na_2CO_3 titration curve	1. Have students measure ml acid(.1N) vs. pH and plot points. Titrate 40 ml Na_2CO_3 solution.
	2. Point out inflection points on standard curve.
	3. $N(.1NHCl) = \frac{ml\ Na_2CO_3}{ml\ HCl} \times (N\ Na_2CO_3)$ $N(.02\ N\ HCl) = N(.1\ NHCl) \times 0.2$
	4. $CaCO_3$ equivalence (mg/ml)=50 X N(HCl)

Module No:	Module Title: Alkalinity Analysis
	Submodule Title:
Approx. Time: 0.5 hours	Topic: Titration of water samples

Instructional Objective:

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

1. Properly titrate a water sample potentiometrically with standard HCl, construct a titration curve, and identify inflection points on the curve.
2. Properly titrate a water sample to a predetermined pH and record appropriate data.
3. Titrate a water sample of low alkalinity to determine total alkalinity.

Instructional Aids:

Transparency Ak5 - sample titration curve

Instructional Approach:

Laboratory practice

References:

Standard Methods, p. 280.

Class Assignments:

Module No: Ak	Topic: Titration of water samples

Instructor Notes:	Instructor Outline:
Transparency Ak-5 Sample titration curve	1. Students perform titration and plot points. Point out inflections on sample curve.
	2. Alternatively have students titrate to predetermined pH(8.3 and 3.7-5.1) and compare with inflection method.
	3. For low alkalinity: a. Use .02 N acid, 100 ml sample b. titrate to 4.5 - record exact pH and volume added(B ml) c. titrate to exactly 0.3 pH units lower, record volume(C ml)
	Total Alkalinity as $CaCO_3$= $\frac{(2B-C) X N(HCl) X 50,000}{ml\ sample}$

Module No:

Module Title:

Alkalinity Analysis

Submodule Title:

Approx. Time:

0.5 hours

Topic:

Calculations

Instructional Objective:

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

1. From titration data, determine the phenophthalein and total alkalinity of a water sample.
2. From the data in 1., determine the hydroxide, carbonate, and bicarbonate alkalinity of a water sample.
3. Properly report the alkalinity of the water sample.

Instructional Aids:

Transparency Ak6 - Alkalinity Relationships

Instructional Approach:

Lecture/discussion

References:

Standard Methods, pp. 280-282

Class Assignments:

Module No: Ak	Topic: Calculations

Instructor Notes:	Instructor Outline:
	1. Alkalinity: Alkalinity($CaCO_3$) mg/l= $\frac{(\text{ml acid}) \text{X} N(HCl) \text{X} 50{,}000}{\text{ml sample}}$ ml acid is that needed to reach phenophthalein alkalinity; and is ml acid needed to reach the total end point.
Transparency Ak-6 Alkalinity relationships	2. Alkalinity types: From data and table determine alkalinity of three types.
	3. In reporting alkalinity, type and method should both be reported.

Exam Questions

Alkalinity Analysis
Definition of alkalinity

1. Write a chemical equation which describes how the hydroxide ion reacts with the hydrogen ion to form water.

2. Alkalinity can be used in water softening along with hardness to determine:
   a. the rustiness of the water
   b. the bacteria level
   c. the relative amounts of lime and soda ash to be used
   d. the potability of the water

3. The pH end point for pherolphthalein alkalinity is:
   a. 8.3
   b. 5.1
   c. 4.5
   d. depends on conditions

4. Which of the following ions is titrated first in an alkalinity determination?
   a. bicarbonate
   b. hydroxide
   c. carbonate

5. The larger the pH value, the __________ the hydrogen ion concentration.

Use of the pH meter

6. The pH meter measures:
   a. color
   b. calcium ion concentration
   c. temperature
   d. hydrogen ion concentration

7. What solution is used to standardize the pH meter?

8. What type of electrode is placed in solution to measure pH?

Safety

9. What device in the lab can be used to wash off acid spilled all over your body?

10. ______________ can be used to protect hands from acid burns.

11. Why should the cord of the pH meter be grounded?

Preparation of Reagents

12. What is the Normality of a Na_2CO_3 solution in which 2.5 g have been dissolved in water for a total volume of 1000 ml?

13. To prepare a solution which is approximately 0.1 N in HCl how many milliliters of concentrated hydrochloric acid(12 N) should be diluted to 1 liter?
    a. 20 ml
    b. 1 ml
    c. 8.3 ml
    d. 100 ml

Standardization of Acid

14. When a Na_2CO_3 solution is titrated with 0.1 N HCl, what instrument is used to determine the points on the titration curve?

15. How many inflection points will be observed in the titration curve of Na_2CO_3?

16. If 20 ml of HCl solution are required to titrate 40 ml of a Na_2CO_3 solution which contains 2.5 g Na_2CO_3 per liter, what is the normality of the HCl solution?

17. What is the $CaCO_3$ equivalence of a 0.1 N HCl solution?

Titration of water samples

18. In the potentiometric titration, the first end point is due to phenolphtalein alkalinity, the second end point is due to ________________ alkalinity.

19. When a water sample is titrated to a predetermined pH, what two pieces of data should be recorded?

20. What concentration acid should be used for titration of low alkalinity?

Calculations

21. If it required 10 ml of acid which is equivalent to 5 mg/ml $CaCO_3$ to titrate to pH 8.3, calculate the phenolphtalein alkalinity of the 50 ml sample in mg/l $CaCO_3$.

22. A sample has a phenolphthein alkalinity of zero(0) and a total alkalinity of 100 mg/l as $CaCO_3$, calculate the bicarbonate alkalinity.

23. Name 3 pieces of data which should be recorded when reporting alkalinity.

ALKALINITY ANALYSIS

EQUIPMENT AND SUPPLIES LIST

1. sodium carbonate Na_2CO_3
2. drying oven
3. calcium chloride
4. dessicator
5. analytical balance
6. weighing bottle
7. 1 liter volumetric flask
8. distilled water
9. 10 ml graduated pipet
10. concentrated hydrochloric acid
11. 100 ml pipet
12. 2 - 50 ml burets
13. pH meter and electrodes
14. pH 7 buffer
15. wash bottle
16. 250 ml beaker
17. 100 ml graduated cylinder
18. bunsen burner, ring stand
19. graph paper
20. 20 ml pipet
21. magnetic stirrer and

Alkalinity Analysis

Laboratory Procedure

I. Preparation of Reagents and Standards

A. Obtain the equipment, supplies, and chemicals listed in the "equipment" handout.

B. Prepare the following solutions:

1. 0.05N Na_2CO_3. In a weighing bottle dry 5g primary standard sodium carbonate for 4 hours in an overn at 250°C. Cool in a dessicator containing $CaCl_2$. Weigh the bottle on an analytical balance. Transfer 2.5g to a 1ℓ volumetric flask and fill to the mark with distilled water. Reweigh the bottle. The mass of the Na_2CO_3 is equal to the difference of the two weighings. Call this A.

2. 0.1N HCl. Pipet 8.3 ml concentrated hydrochloric acid (HCl) into a 1ℓ volumetric flask. Dilute to the mark with distilled water. Mix.

3. 0.02N HCl. Tranfer 200 ml of the 0.1NHCl to a 1 liter volumetric flask. Dilute to the mark with distilled water.

II. Standardizations

A. 0.1NHCl
Fill a 50 ml buret with Na_2CO_3 solution. Fill another buret with 0.1NHCl. Standardize the pH meter with 7.0 pH buffer. Rinse the electrodes. Add 40.00 ml Na_2CO_3 solution to a 250 ml beaker. Add 60 ml distilled water. Insert pH electrodes. With constant stirring add HCl to a pH of 5.0. Remove electrodes, rinse into beaker. Boil contents of beaker for 5 minutes. Allow beaker to cool to room temperature. Titrate further, 0.2 ml at a time. Plot ml vs. pH on graph paper. Determine the inflection point (point of greatest slope). Report total ml required to reach inflection from initial minus finall buret readings. Repeat procedure twice. Calculate the normality from the formala.

$$N=\frac{AxB}{53.0xC}$$

Where: A is the g Na_2CO_3 used.
B is the ml Na_2CO_3 used.
C is the ml acid used.

Calculate the average N.

B. 0.02NHCl
Fill a 50 ml buret with Na_2CO_3 solution. Fill another buret with 0.02N HCl solution. Add 15 ml Na_2CO_3 solution to a 250 ml beaker. Add 65 ml distilled water. Titrate as in A above potentiometrically in triplicate. Record similar data and calculate normality (N) according to the same formula.

III. Potentiometric titration curve

A. Preparation. Fill a 50 ml buret with standardized 0.1N HCl. Rinse the electrodes of a standardized pH meter. Pipet 20 ml well-mixed sample containing 50-200 mg total alkalinity into a 250 ml beaker. Insert electrodes.

B. With constant stirring, add 0.2 ml increments of acid. Record the stabilized pH for each increment as well as the buret reading. Continue to pH 3.7.

C. Prepare graph paper. Label the x-axis "ml acid added" and mark increments from 0 to the final buret reading. Label the y axis "pH" and mark in crements from 0 to 12. Title the graph "Titration curve for Alkalinity Analysis". Plot points recorded in B and connect with a smooth curve. Identify phenolphtalein inflection point (about pH 8.3) and total inflection point (about pH 5.0). Record ml HCl required to reach each end point.

IV. Titration of low alkalinity sample.

A. Transfer 100 ml low alkalinity sample to a 250 ml meaker. Insert pH electrodes. Fill a 10 ml microburet with 0.02N standard HCl. Add acid dropwize with stirring until the pH reads 4.5.

B. Record the exact pH and the exact number of milliliters acid required to reach this pH. Add acid dropwize with stirring to reach a pH value exactly 0.3 units less than the first pH. Record the new buret reading.

V. Calculations

A. Potentiometric titration curve (high alkalinity)

1. To obtain phenolphthalein alkalinity, multiply the ml acid required to reach end point by 50,000 and by the exact acid normality and divide by 20.0, the ml sample used.

2. To obtain total alkalinity, multiply the ml acid required to reach end point by 50,000 and by the exact acid normality and divide by 20.0, the volume sample used.

3. Choose which of the following may be the case: P=0, P less than half T. P equals ½T, P greater than half T, P equals T. Choose the appropriate horizontal row on the data sheet and calculate hydroxide, carbonate, and bicarbonate alkalinity where appropriate. (P equals phenolphthalein alkalinity. T equals total alkalinity).

4. Calculation of low alkalinity: Double the ml titrant required to reach initial pH subtract ml required to reach final pH. Multiply this result by the acid normality and 50,000. Divide by 100, the sample volume.

5. Comment on sample source, possible errors, and other suspected ions persent.

Alkalinity Analysis

Data Sheet

Low Alkalinity
Sample no.__________

High Alkalinity
Sample no.__________

Na_2CO_3 solution

mass of full weighing bottle	________g
mass of weighing bottle with sample removed	________g
mass of sample	________g

Standardization of 0.1N HCl

	I	II	III
final Na_2CO_3 buret	____ml	____ml	____ml
initial Na_2CO_3 buret	____ml	____ml	____ml
ml Na_2CO_3	____ml	____ml	____ml

Titration to inflection

I ml	I pH	II ml	II pH	III ml	III pH

attach titration curves-circle inflection point

ml to inflection:

	I	II	III
final HCl buret readings	____ml	____ml	____ml
initial HCl buret readings	____ml	____ml	____ml
ml acid used	____ml	____ml	____ml
$N=(AxB)/(53xC)$	____N	____N	____N
Average N (HCl)	____N		

Standardization of 0.02N HCl

	I	II	III
final $Na_2\ CO_3$ buret	____ml	____ml	____ml
initial $Na_2\ CO_3$ buret	____ml	____ml	____ml
B ml Na_2CO_3	____ml	____ml	____ml

Titration to inflection

I ml	I pH	II ml	II pH	III ml	III pH

Attach titration curves circle inflection points.

ml to inflection	I	II	III
final HCl buret reading	____ml	____ml	____ml
initial HCl buret reading	____ml	____ml	____ml
ml Acid used	____ml	____ml	____ml
N=(AxB)/(53xC)	____N	____N	____N
Average N(HCl)	____N		

Titration for curve: (attach plotted graph)

ml acid	pH	ml acid	pH	ml acid	pH

ml to reach end point (high alkalinity sample)

phenolphthalein ________ ml A_p
total ________ ml A_t

Titration of low alkalinity sample.

first pH ________
ml required ________ ml B
second pH ________
new buret reading ________ ml C

Calculations

High Alkalinity

phenolphthalein alkalinity
$(A_p x N x 50,000)/20.0 =$ ________ mg/l as $CaCO_3$

total alkalinity
$(A_t x N x 50,000)/20.0 =$ ________ mg/l as $CaCO_3$

Case	OH alkalinity	CO_3^{2-} alkalinity	HCO_3^- alkalinity
P=0	0	0	T
P<½T	0	2P	T-2P
P=½T	0	2P	0
P>½T	2P-T	2(T-P)	
P=T	T	0	0

Bicarbonate alkalinity = ________ (formula) = ________ mg/l $CaCO_3$

Carbonate alkalinity = ________ (formula) = ________ mg/l $CaCO_3$

Hydroxide alkalinity = ________ (formula) = ________ mg/l $CaCO_3$

Low alkalinity:

total alkalinity $= \frac{(2B-C) x N x 50,000}{100.0} =$ ________ mg/l as $CaCO_3$

Comments:

Analyst ____________________ Date ____________________

TRANSPARENCY AK I

Titrations end points

total alkalinity	pH of total alkalinity end point	pH of phenolphthalein end point
30 mg/l	5.1	8.3
150 mg/l	4.8	8.3
500 mg/l	4.5	8.3
silicates or phosphates	4.5	8.3
complex systems	3.7	8.3

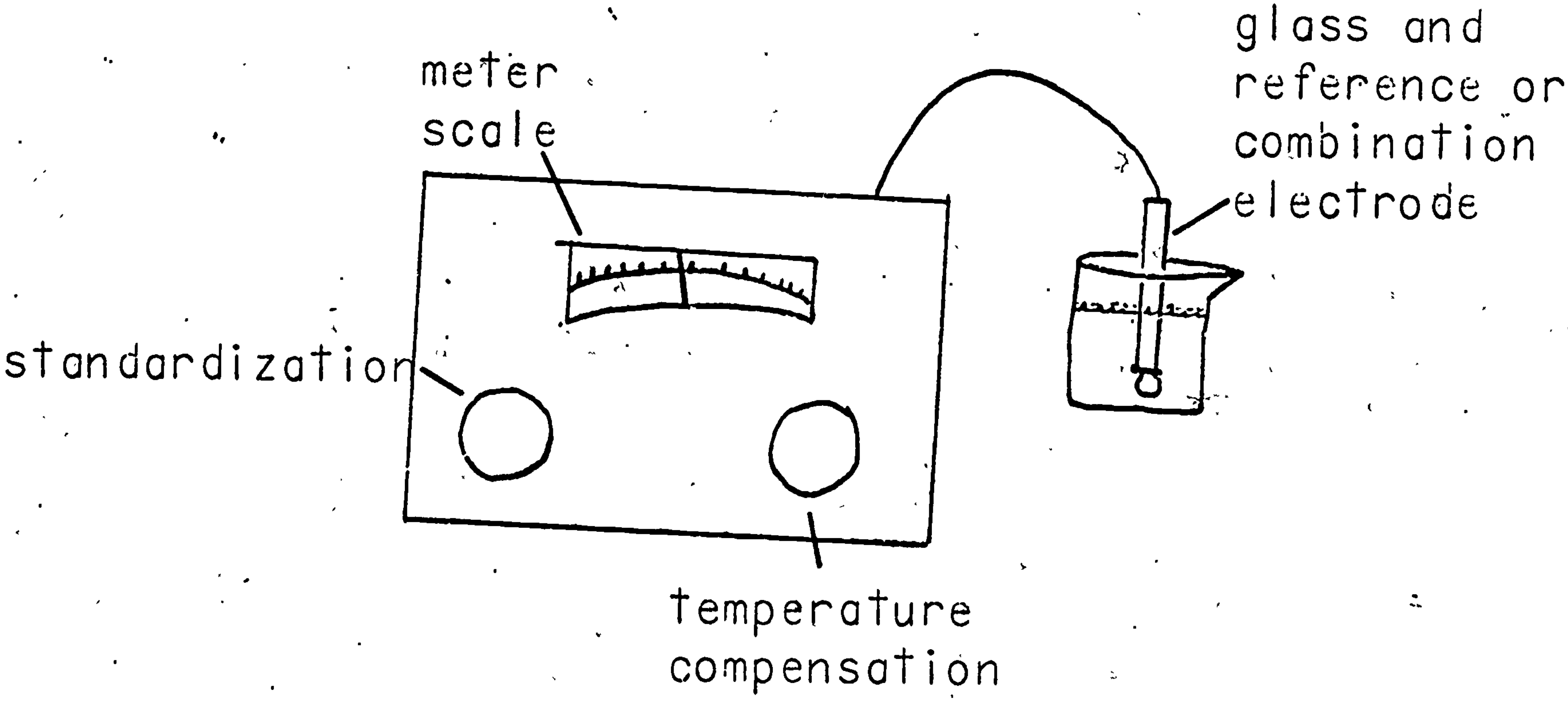
TRANSPARENCY AK2

Relation of pH to Hydrogen and Hydroxide Ion Concentration (Molarity)

pH	OH^-	H^+	
1	10^{-13}	10^{-1}	ACIDIC
2	10^{-12}	10^{-2}	
3	10^{-11}	10^{-3}	
4	10^{-10}	10^{-4}	
5	10^{-9}	10^{-5}	
6	10^{-8}	10^{-6}	
7	10^{-7}	10^{-7}	
8	10^{-6}	10^{-8}	BASIC
9	10^{-5}	10^{-9}	
10	10^{-4}	10^{-10}	
11	10^{-3}	10^{-11}	
12	10^{-2}	10^{-12}	
13	10^{-1}	10^{-13}	
14	1	10^{-14}	

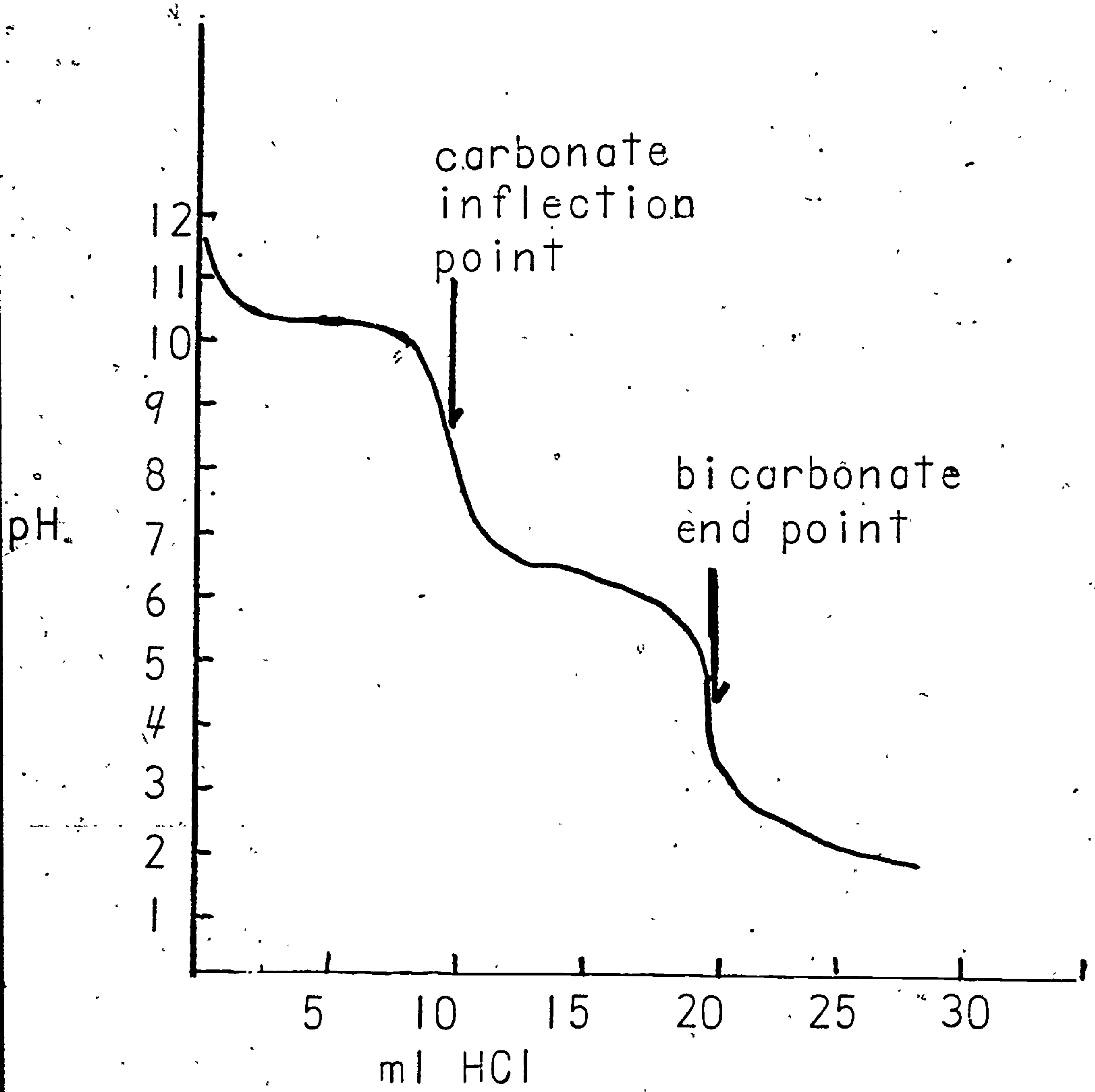
TRANSPARENCY AK3

Diagram of pH Meter



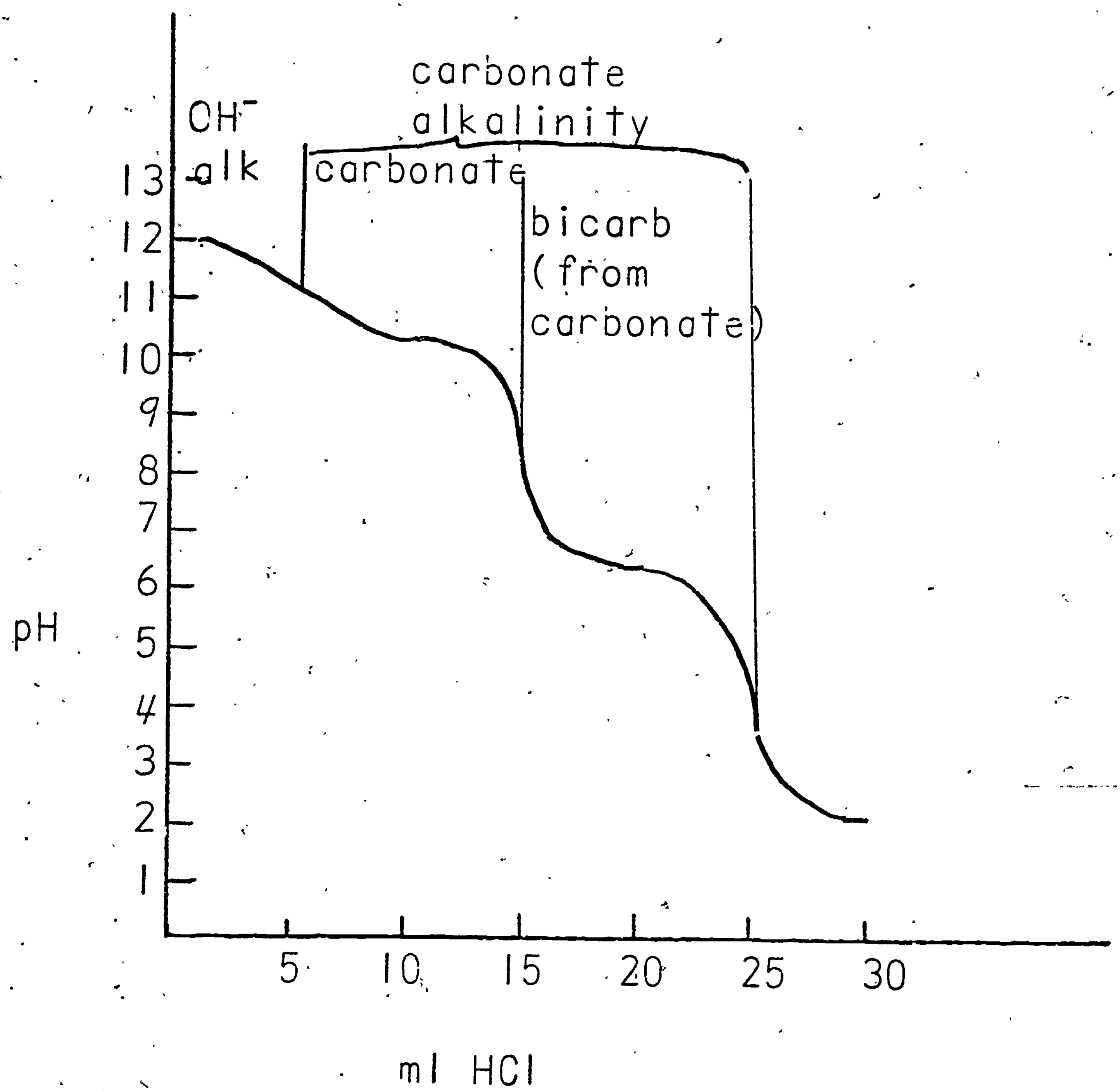
TRANSPARENCY AK4

Sample Na_2CO_3 Titration Curve



TRANSPARENCY AK5

Water Sample Titration Curve



TRANSPARENCY AK6

Alkalinity Relationships

P= phenolphthalein alkalinity(first inflection)

T= total alkalinity(second inflection)

	OH^- alkalinity	HCO_3^- alkalinity	CO_3^{2-} alkalinit
P=0	0	0	T
P 1/2T	0	2P	T-2P
P=1/2T	0	2P	0
P 1/2T	2P-T	2(T-P)	0
P=T	T	0	0