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AUTHOR Bonte, John L.; Davidson, Arnold C.
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ABSTRACT

This document is an instructional module package prepared in objective form for use by an instructor familiar with the titremetric method and the nomographic method of determining free carbon dioxide concentrations of a water sample. Included are objectives, an instructor guide, student handouts, and transparency masters. A video tape is also available from the author. This module considers chemistry and principles of dissolved carbon dioxide, the preparation of standards and reagents, calculation of free carbon dioxide and the nomographic determination of free carbon dioxide. (Author/RH)

FREE CARBON DIOXIDE

Training Module 5.245.2.77

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

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September, 1977

Module No:

Module Title:

Free Carbon Dioxide

Submodule Title:

Approx. Time:

4.0 hours

Topic:

Summary

Instructional Objective:

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

1. Determine the pH, temperature, and filtrable residue content of a water sample.
2. Calculate free CO_2 level by the nomographic method.
3. Determine the free CO_2 level by the titrimetric method.

Instructional Aids:

Transparency CD1 - CD4- Surface water videotape

Instructional Approach:

Lecture, demonstration, discussion, laboratory practice, videotape viewing.

References:

1. Standard Methods for the Examination of Water and Wastewater, 14 edition.
2. Operator's manual for pH meter.

Class Assignments:

Module No:	Module Title: Free Carbon Dioxide
	Submodule Title:
Approx. Time: 0.5 hours	Topic: Chemistry of dissolved CO_2

Instructional Objective:

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

1. Describe how free CO_2 is related to corrosion and water hardness.
2. List the three forms of CO_2 in water.
3. Qualitatively relate the three forms to pH.

Instructional Aids:

Transparency CD1- three forms of CO_2 and pH.

Instructional Approach:

Lecture/discussion

References:

Standard Methods p. 293, 61-63.

Class Assignments:

Module No: CD	Topic: Chemistry of dissolved CO_2

Instructor Notes:	Instructor Outline:
	1. The gas, carbon dioxide reacts with pure water to form carbonic acid which will lower pH, CO_2 may react with CaO and other metal oxides to form $CaCO_3$ and other carbonates. Since $CaCO_3$ is only slightly soluble, the free CO_2 level and pH will determine the amount of hardness which will precipitate and whether a protective $CaCO_3$ coating will be in water mains.
	2. Forms of CO_2 a. $H_2CO_3 = CO_2 + H_2O$ (Free CO_2) b. HCO_3^- bicarbonate c. CO_3^{2-} carbonate
Transparency CD-1 Forms of CO_2 and pH	3. pH and CO_2 a. low pH, H_2CO_3 b. intermediate pH, HCO_3^- c. high pH, CO_3^{2-}

Module No:	Module Title: Free Carbon Dioxide
	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 appropriate situation.
3. Describe hazards involved with the chemicals used in determining CO_2.

Instructional Aids:

Laboratory safety rules handout.

Instructional Approach:

Lecture/demonstration

References:

Basic laboratory skills module.

Class Assignments:

Module No: CD	Topic: Safety
Instructor Notes:	Instructor Outline:

1. Point out to students the location of various safety devices and their use.

2. Safety glasses should be worn during the preparation of reagents and during titration.

3. The Na_2CO_3 solutions are basic and may cause chemical burns.

Module No:	Module Title: Free Carbon Dioxide
	Submodule Title:
Approx. Time: .5 hours	Topic: Principles of CO_2 measurement

Instructional Objective:

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

1. List two methods for determining free CO_2.
2. List data needed for nomographic determination of CO_2.
3. Describe qualitatively the titrimetric method of CO_2 determination.

Instructional Aids:

Surface water analysis videotape.

Instructional Approach:

Lecture, discussion, videotape viewing

References:

Standard Methods, pp. 293 - 301.

Class Assignments:

Module No: CD	Topic: Principles of CO_2 measurement

Instructor Notes:	Instructor Outline:
Surface water analysis, Video tape - middle section	1. Show video tape - answer questions 2. Methods a. nomographic b. titrimetric 3. Nomographic data a. temperature b. pH c. total filtrable residue d. total alkalinity 4. Titration: The H_2CO_3 is consumed completely and converted to HCO_3^- by each mole of NaOH or Na_2CO_3

Module No:	Module Title: Free Carbon Dioxide
	Submodule Title:
Approx. Time: 0.5 hours	Topic: pH measurement

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 CD2 - Diagram of pH meter.

Instructional Approach:

Laboratory practice

References:

Operator's manual for pH meter used.

Class Assignments:

Module No: CD	Topic: pH measurement
Instructor Notes:	Instructor Outline:
Transparency CD-2 Diagram of pH meter	1. Point out parts of pH meter a. meter b. calibration knob c. electrodes 2. Standardize against pH 7 buffer 3. Measure pH of any water solution

Module No:

Module Title:
Free Carbon Dioxide

Submodule Title:

Approx. Time:
0.5 hours

Topic:
Filtrable Residue

Instructional Objective:

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

1. Determine the concentration of filtrable residue in a water sample.
2. Differentiate between filtrable and non-filtrable residue.

Instructional Aids:

Transparency CD3 - filter apparatus

Instructional Approach:

Laboratory practice

References:

Standard Methods p.92

Class Assignments:

Module No: CD	Topic: Filtrable residue

Instructor Notes:	Instructor Outline:
Transparency CD-3 Filter apparatus	1. Filtrable residue a. filter sample through fritted glass b. evaporate filtrate to dryness c. weigh residue d. mg/l=mg residue/liters of sample 2. Residue: a. filtrable - dissolved b. non-filtrable - suspended

Module No:

Module Title:
Free Carbon Dioxide

Submodule Title:

Approx. Time:
0.25 hours

Topic:
Nomograph Method

Instructional Objective:

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

1. Determine the free CO_2 level of a water sample from pH, residue, temperature and total alkalinity using a nomograph.
2. Determine the various types of alkalinity from pH, residue, temperature and total alkalinity.

Instructional Aids:

Transparency CD4- Free CO_2 Nomograph

Instructional Approach:

Lecture/discussion.

References:

Standard Methods p. 294-298.

Class Assignments:

Module No: CD	Topic: Nomograph Method

Instructor Notes:	Instructor Outline:
Transparency CD-4 Free CO_2 Nomograph, ruler	1. Describe steps in using nomograph by using data as follows: a. align temperature, 25° C b. total filtrable residue, 200 mg/l c. determine P_1 d. align pH, 7.5 e. bicarbonate alkalinity, 200 mg/l f. determine P_2 g. align P_1, P_2 h. read CO_2: 11.0 mg/l 2. The same procedure should be used on their data from residue, pH, and alkalinity determinations. 3. Point out that the forms of alkalinity can also be determined by nomograph given, pH, temperature, total alkalinity, residue.

Module No:	Module Title: Free Carbon Dioxide
	Submodule Title:
Approx. Time: 0.25 hours	Topic: Preparation of Standards and reagents

Instructional Objective:

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

1. Prepare a standard Na_2CO_3 solution for free CO_2 determination.
2. Properly collect a water sample for CO_2 analysis.

Instructional Aids:

Instructional Approach:

Laboratory practice

References:

Standard Methods p. 298-300.

Class Assignments:

Module No: CD	Topic: Preparation of standards and reagents

Instructor Notes:	Instructor Outline:
1. Na_2CO_3 solution: 2.407 g dissolved in water to make 1 liter. Distilled water should be boiled Na_2CO_3 should be dry.	1. 0.0454 N Na_2CO_3 preparation 2. Collect sample in 500 ml Pyrex bottle - no trapped air. Syphon sample into graduated cylinder with overflow.

Module No:	Module Title: Free Carbon Dioxide
	Submodule Title:
Approx. Time: 0.5 hours	Topic: Titration and calculation of CO_2

Instructional Objective:

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

1. Titrate the sample with standard base to a predetermined end point or inflection point.
2. Calculate the concentration of free CO_2 from titration data.
3. List precautions to be taken in CO_2 analysis.

Instructional Aids:

Instructional Approach:

Laboratory practice

References:

Standard Methods pp. 298-300.

Class Assignments:

Module No: CD	Topic: Titration and calculation of CO_2

Instructor Notes:	Instructor Outline:

1. Titration
   a. add 5 drops phenolphthalein
   b. if solution turns red - no CO_2
   c. if colorless add Na_2CO_3 solution from a buret, stir with glass rod until pink color persists, record ml Na_2CO_3 used.
   d. repeat but run in entire ml Na_2CO_3 from c. above. If necessary add more titrant to end point.

2. Calculation:
   a. red immediately CO_2=0
   b. $mg/l(CO_2) = \frac{(ml\ Na_2CO_3) X (.0454) X 22{,}000}{ml\ sample}$

3. Precautions:
   a. glass-wear must be clean and free of acid or base residue
   b. titrate rapidly to prevent loss of CO_2 to the atmosphere during titration.

Exam Questions

Free Carbon Dioxide
Chemistry of Dissolved CO_2

1. If carbon dioxide is dissolved in distilled water will the solution be acidic or basic?

2. Three forms of CO_2 in water are free CO_2, bicarbonate, and _____.

3. At extremely high pH, CO_2 is mostly in the _____ form.

Safety

4. Chemicals splashed in the eye can be removed at the _____.

5. _____ will prevent the spillage of chemicals on the hands.

6. Na_2CO_3 will not "burn" when spilled on skin. True or False

Principles of CO_2 Measurement

7. Two methods of determining free CO_2 are the nomographic method and the _____ method.

8. The following data are needed for the nomographic method: pH, alkalinity, temperature, and _____.

9. At the end point of the titrimetric method, all the free CO_2 will be converted to _____ ion.

pH Measurement

10. The pH meter measures _____.

11. The pH meter is standardized with a _____ solution.

12. The pH of the solution is read on the _____ of the pH meter.

Filtrable Residue

13. A 50 ml sample contains 1.2 grams of residue. Calculate the concentration of residue in mg per liter.

14. Does filtrable residue include dissolved salts?

Nomograph Method

15. How many pivot lines are required for the determination of free CO_2?

16. Besides free CO_2, what else can be determined from a nomograph, residue, temperature, total alkalinity, and pH?

Preparation of STandards and Reagents

17. How should the water which is used for the preparation of titrant

be treated?

18. Why should care be taken to exclude air from a sample taken for free CO_2 analysis?

Titration and Calculation of CO_2

19. What is the color change in the CO_2 titration?

20. If 5.0 ml of 0.0454 N Na_2CO_3 is required to titrate a 100 ml water sample, what is the concentration of CO_2 in the sample?

21. Briefly describe how CO_2 is prevented from escaping or entering the water sample during collection or sampling.

FREE CO_2

EQUIPMENT AND SUPPLIES LIST

1. Free CO_2 nomograph (or as transparency CD_4)
2. thermometer
3. filter apparatus, filter flask, holder, glass fritted crucible
4. nickel crucible
5. oven
6. analytical balance
7. pH meter
8. pH 7 buffer
9. alkalinity test kit
10. sodium carbonate
11. distilled water
12. 2000 ml beaker
13. 1 liter volumetric flask
14. ringstand, ring, bunsen burner
15. 2 - 1 liter bottle
16. 100 ml graduated cylinder
17. rubber tubing
18. phenolphthalein indicator solution
19. 25 ml buret
20. glass stirring rod

Free Carbon Dioxide

Laboratory Procedure

I. Nomographic Method

A. Obtain a "free CO_2" nomograph (transparency CD4 reproduced as a hand-out), a ruler and the following data for your sample.
   1. temperature
   2. total filtrable residue
   3. pH
   4. bicarbonate alkalinity

B. Sample data

data	1	2	3
temperature	25°C	25°C	12°C
total filtrable residue	700 mg/l	0 mg/l	200 mg/l
pH	8.5	7.0	6.0
bicarbonate alkalinity	300 mg/l	10 mg/l	2 mg/l

C. To use nomograph, (also: copies of the nomograph and nomographs for alkalinity may be obtained from The American Water Works Association, 666 West Quincy Ave, Denver, Colorado 80235.) Align temperature and total filtrable residue which determines P_1 point on line. Align pH and bicarbonate alkalinity which determines P_2 on line 2. Align P_1 and P_2 and read free CO_2 on each line. Using this procedure find free CO_2 for sample data 1, 2, 3, and for the sample provided by the student.

II. Titrimetric Method

A. Prepare a standard sodium carbonate solution. Add 2.407g. Na_2CO_3 to a 1 liter volumetric flask. Boil 1500 ml distilled water for 5 minutes; allow to cool. Fill the volumetric flask to the mark with this water. Label as: 0.0454 N sodium carbonate. Place in a bottle protected from atmospheric CO_2.

B. Collet a sample and analyze as soon as possible after collection by directing discharge into bottom of collection bottle. Protect from out-gassing or in-gassing. Syphon sample into 100 ml graduated cylinder allowing overflow. Flick sample to throw off sample above 100 ml mark.

C. Add 5-10 drops phenolphthalein indicator. Add the Na_2CO_3 solution to a 25 ml buret. Record the initial buret reading. Dropwize, add Na_2CO_3 to the graduated cylinder with stirring until a pink color persists for 30 seconds. Record the buret reading. The difference in the two readings is the amount of titrant added.

D. Calculate the free CO_2 level by multiplying the ml Na_2CO_3 added by 9.988. Comment on collection, storage, or other irregularities.

Data Sheet for Free Carbon Dioxide

I. Nomographic Method
Sample data

	1	2	3
Free CO_2	_____ mg/l	_____ mg/l	_____ mg/l

Data from sample no _____

temperature _____ °C

Filtrable residue _____ mg/l

pH _____

Bicarbonate alkalinity _____ mg/l

Calculated free CO_2 _____ mg/l

II. Titrimetric Method

Sample no. _____

initial buret reading _____ ml

final buret reading _____ ml

ml Na_2CO_3 added _____ ml (A)

Free CO_2 = 9.988 • A = _____ mg/l

Comments:

Analyst _____ Date _____

TRANSPARENCY CD1

Three forms of CO_2 and pH

Carbonic acid(free CO_2)- low pH, acid

H_2CO_3

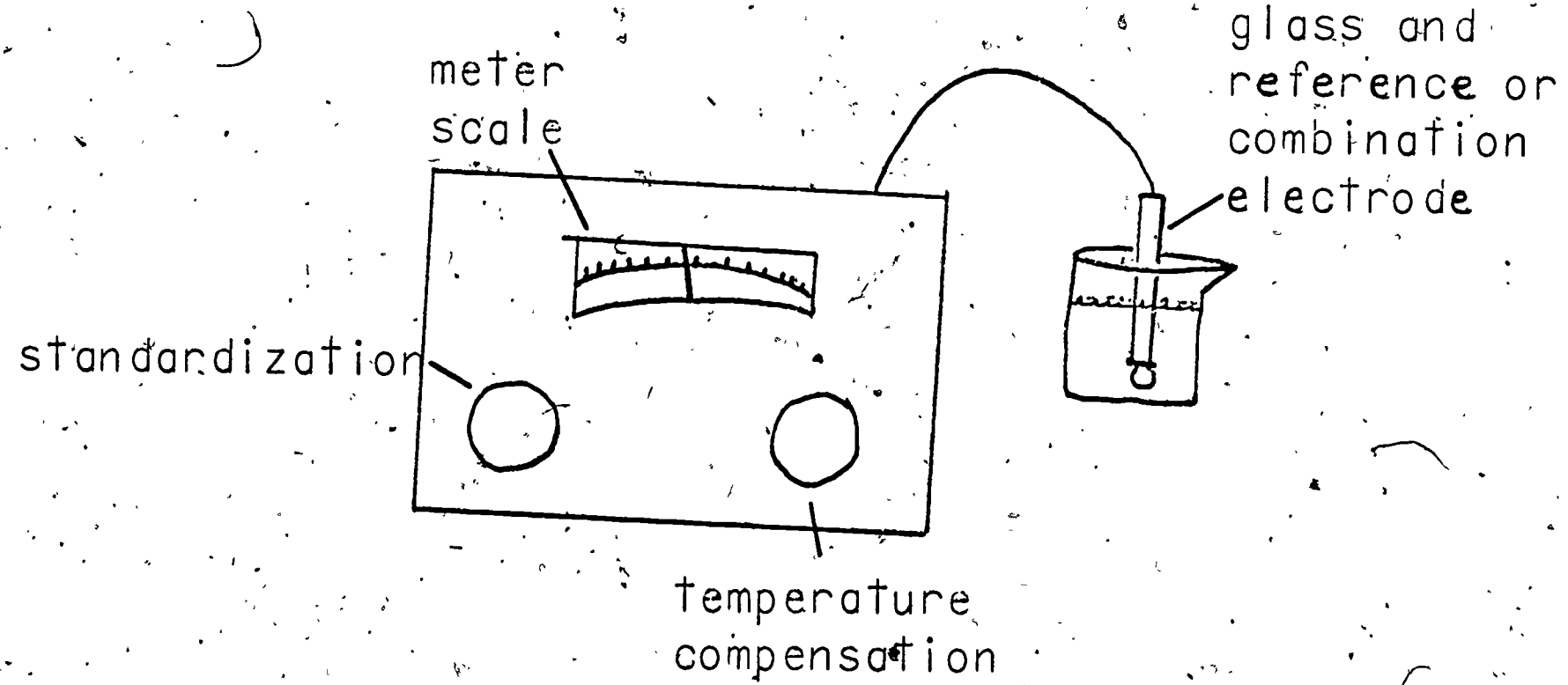
Bicarbonate-intermediate pH, neutral

HCO_3^-

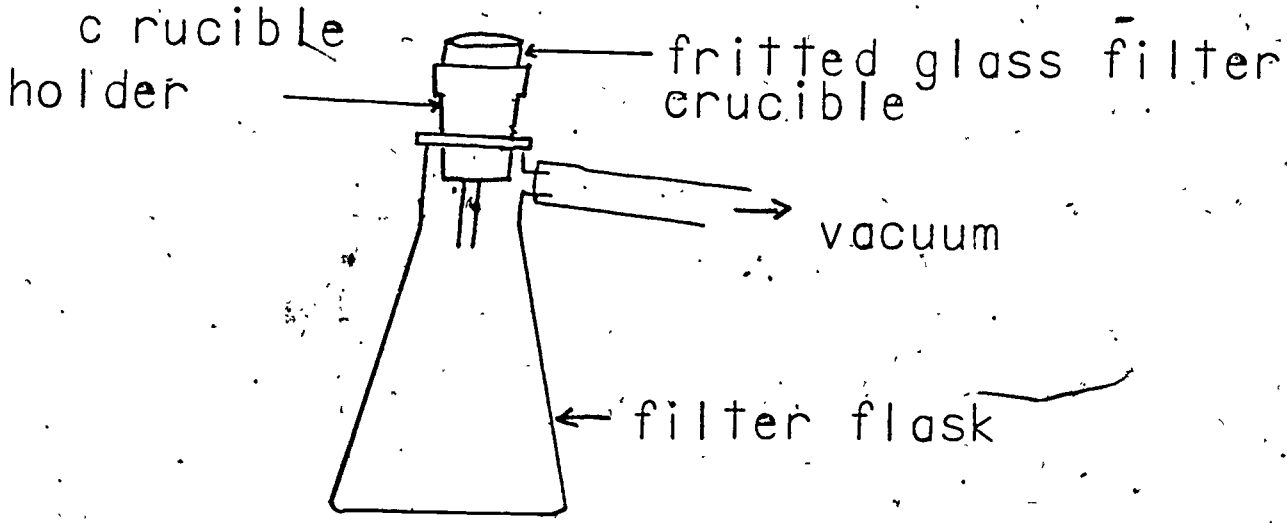
Carbonate- high pH, basic

CO_3^{2-}

TRANSPARENCY CD2
Diagram of pH Meter



TRANSPARENCY CD3
Filter Apparatus



TRANSPARENCY CD4

Free CO_2 Nomograph