



UNITED STATES MARINE CORPS
Base Maintenance Division
Marine Corps Base
Camp Lejeune, North Carolina 28542

C.J.

IN REPLY REFER TO
MAIN/FEC/rn
11345
16 Nov 1983

From: Director, Utilities Branch
To: Wastewater Treatment Plant Operators
Subj: Wastewater Sampling and Analysis

1. This booklet contains revised procedures for sampling and analysis of wastewater at our sewage treatment plants.
2. Effective immediately, all operators shall become thoroughly familiar with the contents of this booklet and shall strictly follow the procedures outlined.
3. Any questions that may arise should be directed to the shift leader or immediate supervisor. In addition, NREAD personnel will visit plants periodically to check sampling/analysis procedures and provide assistance as required.

F. E. Cone
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JENCO PH METER OPERATIONS

DAILY STANDARDIZATION METHOD

8/4 SHIFT WILL STANDARDIZE PH METER DAILY. ONLY 8/4 SHIFT WILL HAVE TO DO THIS UNLESS THE STAND LED IS FLASHING.

- 1) CUT THE PH METER ON, MAKE SURE THE AUTOLOCK LED IS ON.
- 2) RINSE THE PH & TEMP. ELECTRODE IN DISTILLED WATER
- 3) INSERT BOTH PROBES INTO FRESH PH 7 BUFFER.
- 4) PRESS THE "STAND" BUTTON, (THE WAIT LED WILL FLASH)
- 5) WHEN THE SLOPE LED STARTS FLASHING REMOVE THE PH & TEMP. PROBES FROM THE PH 7 BUFFER, RINSE BOTH PROBES WITH DISTILLED WATER.
- 6) INSERT BOTH PROBES INTO SECOND PH BUFFER (SECOND BUFFER WILL BE EITHER PH 4 OR 10 DEPENDING UPON USUAL PH READING OF SAMPLES. I.E. IF YOUR USUAL SAMPLE PH IS UNDER 7.0 USE BUFFER 4.0, IF YOUR USUAL SAMPLE PH IS ABOVE 7.0 USE BUFFER 10.0).
- 7) PRESS THE SLOPE BUTTON, THE WAIT LED WILL FLASH.
- 8) WHEN THE WAIT LED STOPS FLASHING TAKE THE PROBES OUT OF THE PH BUFFER AND RINSE WITH DISTILLED WATER.

THE PH METER IS NOW STANDARDIZED AND READY TO USE.

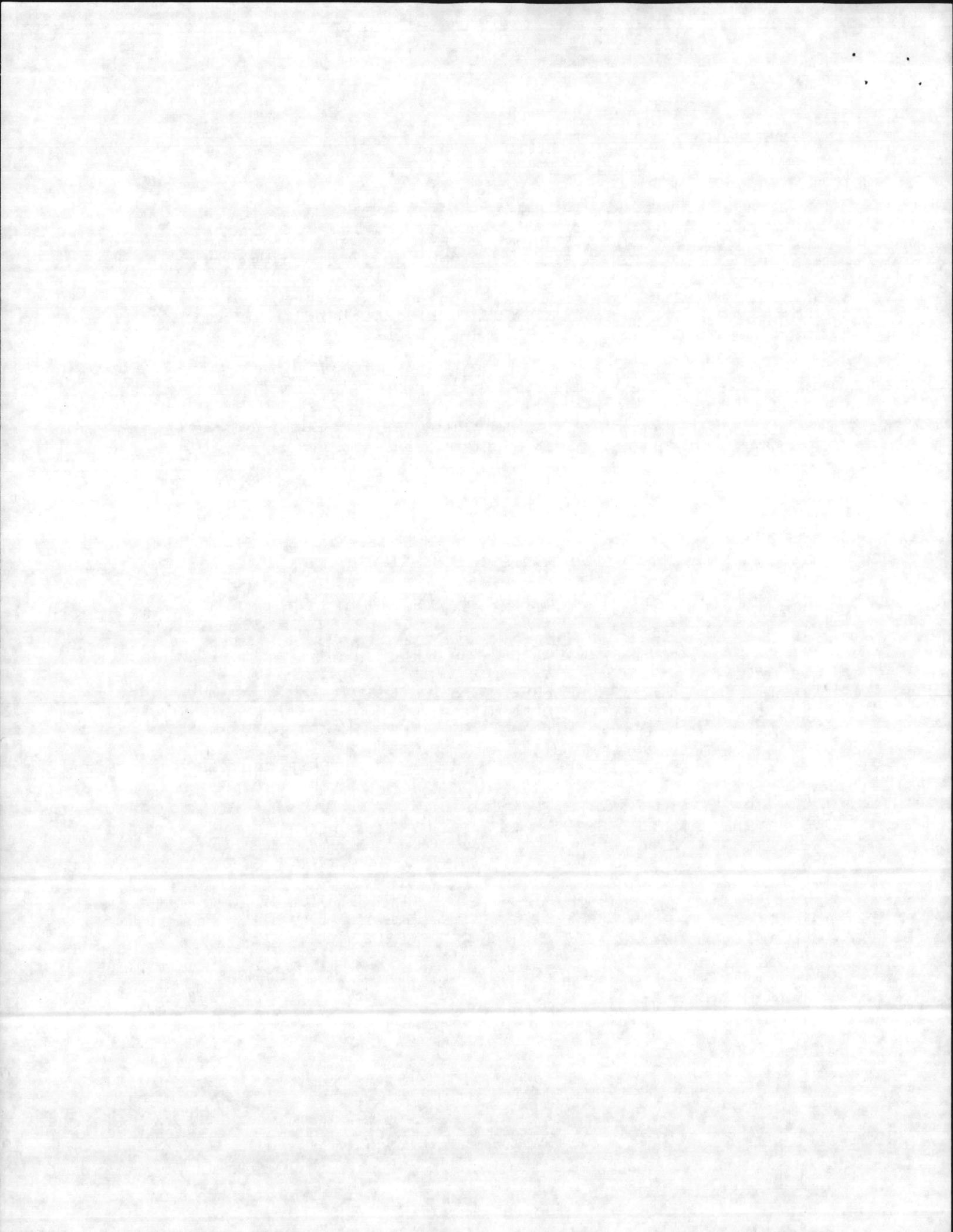
USING THE METER TO TAKE A PH READING.

- 1) IF THE PH METER IS OFF PRESS THE ON BUTTON, IF THE STAND LED IS FLASHING REFER TO INSTRUCTIONS ABOVE.
- 2) RINSE BOTH PROBES WITH DISTILLED WATER, INSERT BOTH PROBES INTO A BEAKER OF SAMPLE.
- 3) PRESS THE MEASURE BUTTON, (THE WAIT LED WILL FLASH, WHEN THE WAIT LED STOPS FLASHING YOU CAN TAKE YOUR READINGS). THE TOP DISPLAY IS THE PH READING, THE BOTTOM DISPLAY IS THE TEMP. IN CENTIGRADE.
- 4) CUT THE PH METER OFF.

THE PH METER SHOULD ALWAYS BE IN THE pH/AUTOLOCK MODE. IF PH IS NOT SHOWING IN BOTTOM RIGHT-HAND CORNER OF TOP DISPLAY AND THE AUTOLOCK LED IS NOT ON PRESS THE MODE BUTTON UNTIL THEY SHOW (SHOULD NOT BE MORE THAN THREE PRESSES).

IF AN ERROR MESSAGE APPEARS PRESS THE CLEAR BUTTON AND START OVER AGAIN.

IF YOU HAVE ANY PROBLEMS SEE SUPERVISOR.



pH Buffer Values for Varying Temperatures

4.01 pH Buffer

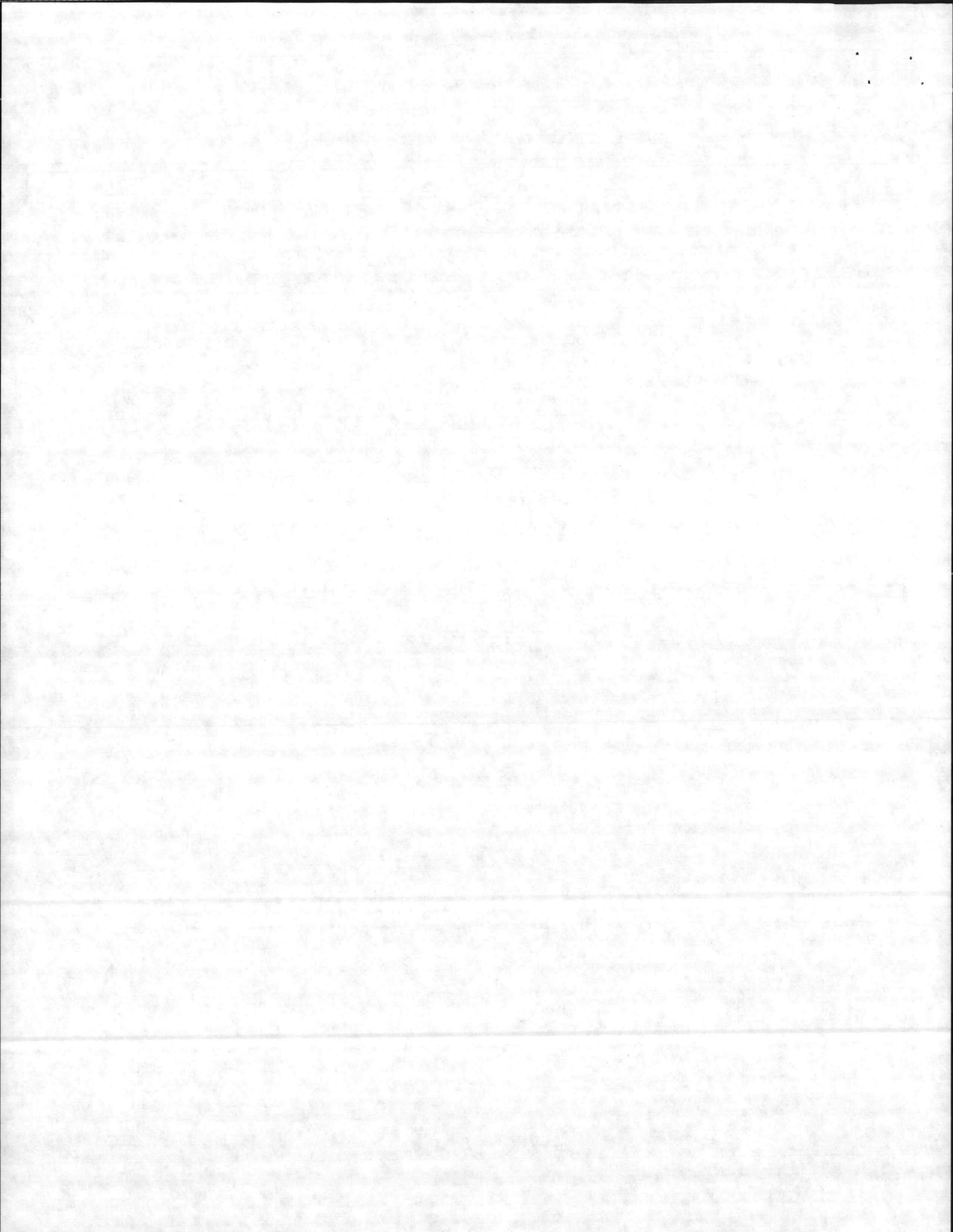
Temperature	pH
Range (°C)	Value
0-49	4.0
50-77	4.1
78-95	4.2

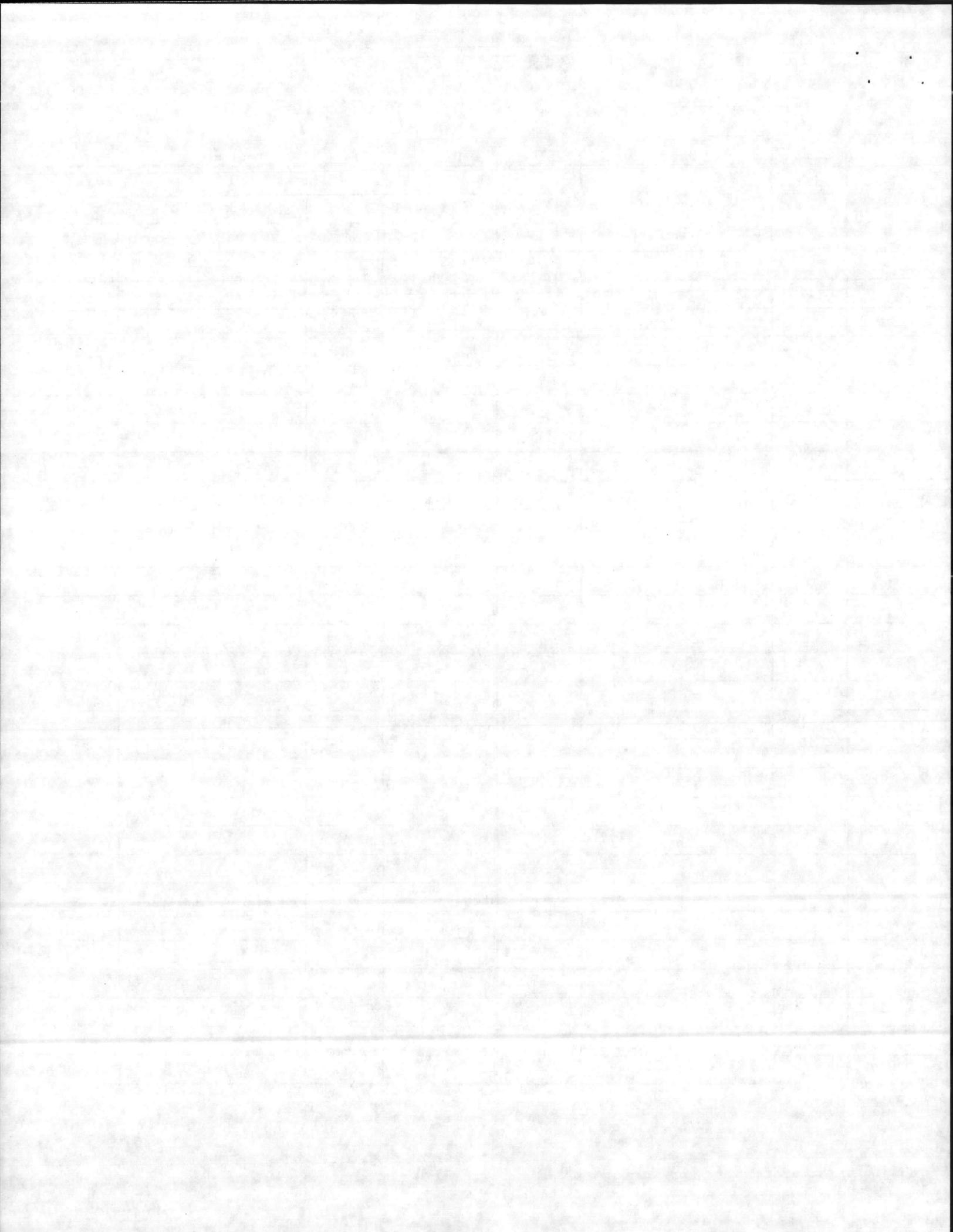
7.00 pH Buffer

Temperature	pH
Range (°C)	Value
0-17	7.1
18-95	7.0

9.18 pH Buffer

Temperature	pH
Range (°C)	Value
0-3	9.5
4-8	9.4
9-17	9.3
18-27	9.2
28-42	9.1
43-61	9.0
62-90	8.9
91-95	8.8





Total Residual Chlorine Test-Wastewater Treatment

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DPD Tablet Comparator Method

NO LONGER
USES THIS
METHOD.

I. Sampling

- A. Collect a representative sample in both precision tubes. For best results, rinse the tubes two or three times with sample to be tested.
- B. Perform the test as soon as possible after sample collection.

II. Equipment and Reagents-Supplied by Utilities Branch, BMaintDiv

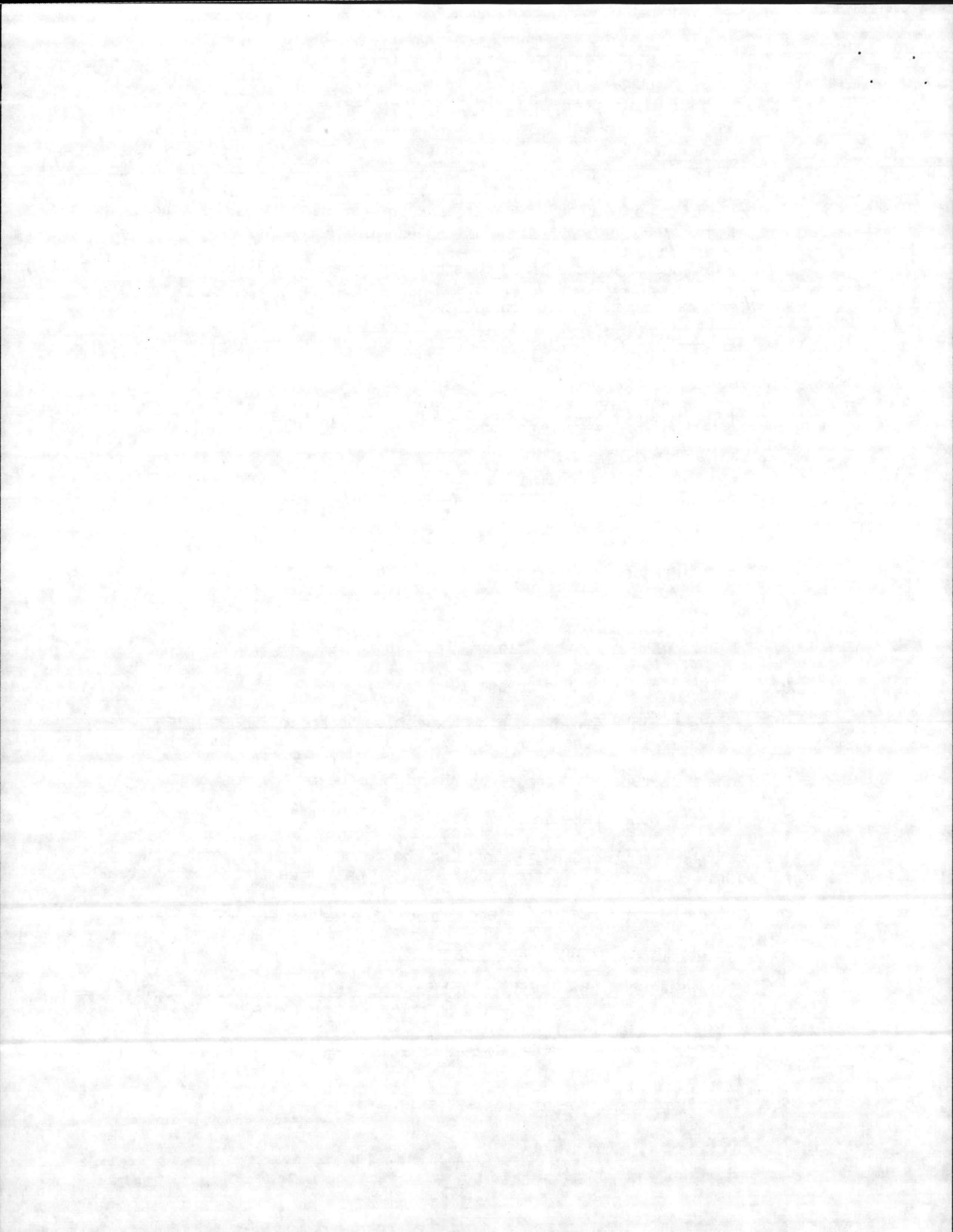
- A. Hellige Color Comparator
- B. Precision Tubes
- C. Hellige DPD Chlorine Color Disc, 0.2-4.0 range
- D. Hellige DPD Tablets #4

III. Equipment and Reagents-Supplied by Quality Control Lab, Envir. Br., NREAD

- A. Glass Stirring Rods
- B. Distilled Water

IV. Procedure

- A. Check to make sure the Chlorine Disc is in the comparator.
- B. Fill both sample tubes to the 10 ml mark.
- C. Add one tablet, avoid touching the tablet to one of the sample tubes and mix. If necessary, use the stirring rod to help break up the tablet.
- D. Place this tube with tablet in the right side of the comparator.
- E. Place the other tube in the left side.
- F. Read the ppm chlorine by comparing the colors of the chlorine disc to the sample within 3 minutes. If the reading is 3.0 ppm or less proceed to step 13.
- G. If the chlorine reading is above 3.0 ppm, collect another sample.
- H. Fill both sample tubes to the 5 ml mark.
- I. Add distilled water to both, to bring the level to the 10 ml mark. Mix the samples.
- J. Add one tablet to one tube as in step C. Place this tube in the right side.
- K. Place the other tube in the left side.



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L. Read the ppm chlorine by comparing colors as in step F. Take the ppm reading and double it for the chlorine reading.

M. Record the ppm residual chlorine in the log.

N. Reprint daily averages to Quality Control Laboratory by the 3rd day of following month.

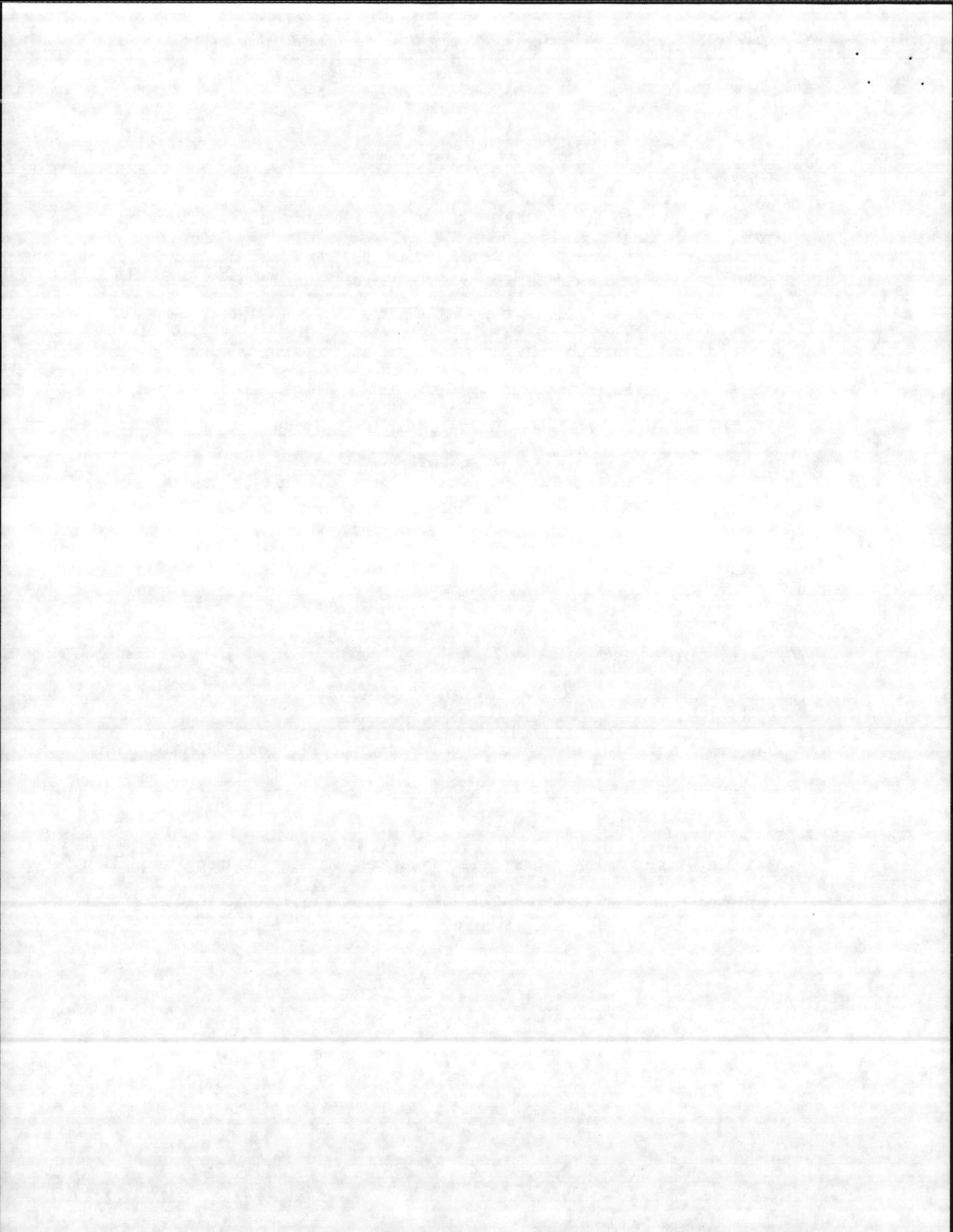
Coliform Sampling Procedure-Wastewater Treatment (See note A)

I. Sampling Steps

- A. Get the sterile coliform bottle and the coliform sampler (do not confuse with the plastic #3 sampler).
- B. Place the bottle in the sampler and CAREFULLY remove the lid. (See Note B)
- C. Avoid touching the inside of the bottle lid and the mouth of the bottle.
- D. Go to the end of the chlorine contact chamber, location labelled #3.
- E. Hold the bottle so as to catch some effluent as it goes over the weir.
- F. Fill the bottle almost full. Leave an air space in the bottle.
- G. CAREFULLY replace the lid tightly and remove from sampler.
- H. On the blank label on the bottle enter the following information:
 1. Date
 2. Time
 3. Your name
 4. Chlorine residual
- I. Place the sample bottle in the refrigerator until its time to carry it to the lab.
- J. Place the sample bottle on the ice while it is being carried to the lab.
- K. At the lab, place it in the refrigerator.

II. Notes

- A. A sample will be taken on schedule, no matter what the effluent looks like.
- B. Sample bottles contain Sodium Thiosulfate to neutralize chlorine. Do not rinse out. Additional bottles are available at WQCL if accidents occur.



Composite Sampling Procedure-Wastewater Treatment (See note 1)
for Influent/effluent

I. Sampling Steps

A. Once every hour, get the correct sampler (I,II, or III) for the correct sample location.

B. Clean it out, if necessary.

C. Go to the sample location.

D. Rinse the sampler 3 times with the sample.

1. Influent (I)-hold the sampler under the water level such that it is not dragging the bottom of the chamber or just skimming the top.

2. Unchlorinated Effluent (II)-Dip down into the chamber, being careful not to drag from the bottom or skim from the top.

3. Chlorinated Effluent (III)-Collect the sample as it comes over the weir.

E. Using the flow rate, determine the sample size from the sample size chart prepared for each plant.

F. Stir up the sample and measure out the correct sample size and add into the correct sample jar kept in the refrigerator.

G. Rinse out the sampler and measurer (graduated cylinder).

H. Record on the label that is on the lid of the bottle the following information (See note below):

1. The time of collection of the first sample.

2. Your last name

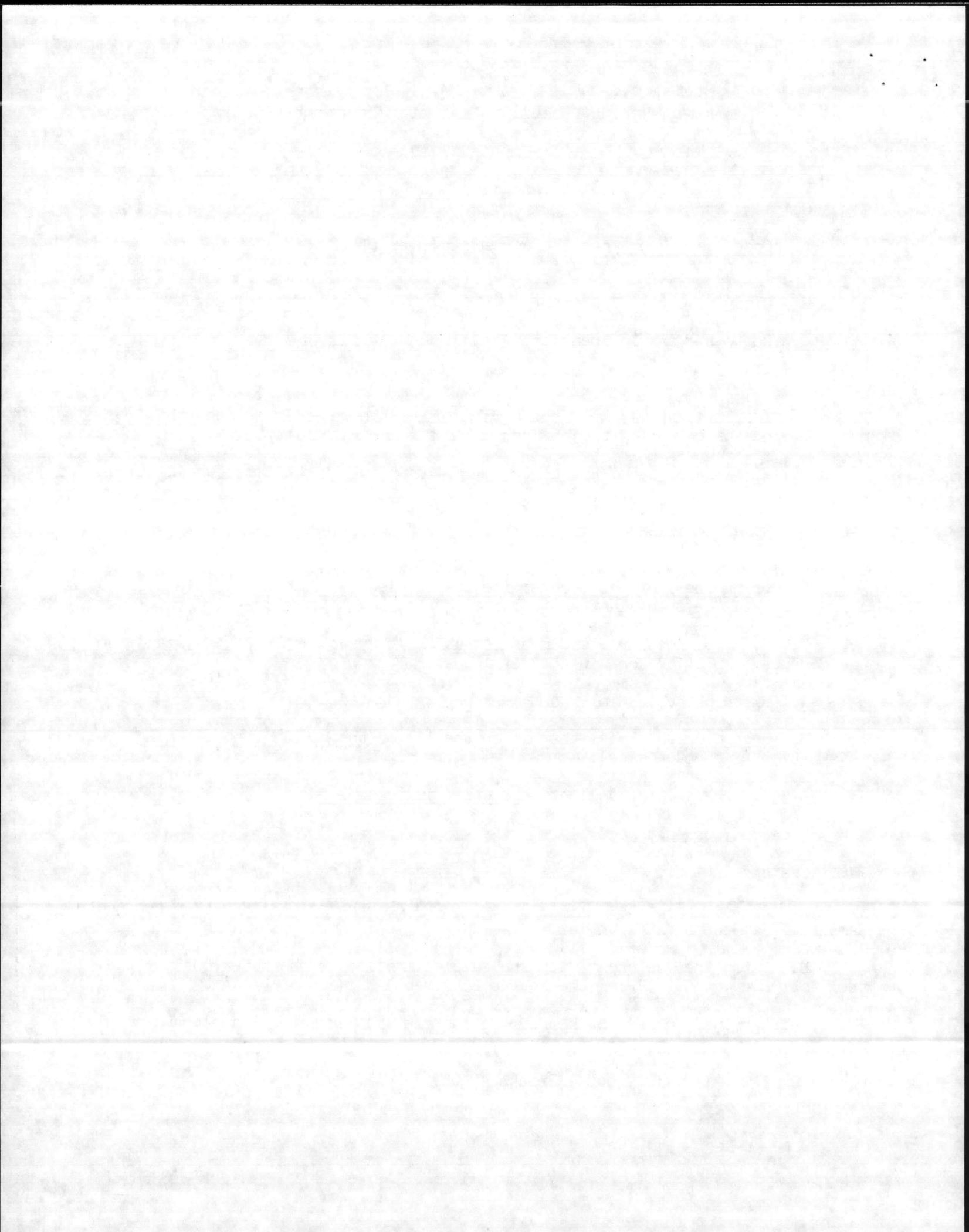
II. Transportation

A. At the end of the sampling period (24 or 8 hours), CAREFULLY place the samples in an ice chest with ice.

B. Carry the ice chest with the samples and ice to the Water Quality Control Lab, Bldg. #65. (Bring the key to BLDG. #65).

C. Carefully place in the refrigerator in the laboratory sample and supply room.

D. Leave the empty bottle racks at the laboratory.



E. Pick up the next set of sample bottles, if necessary.

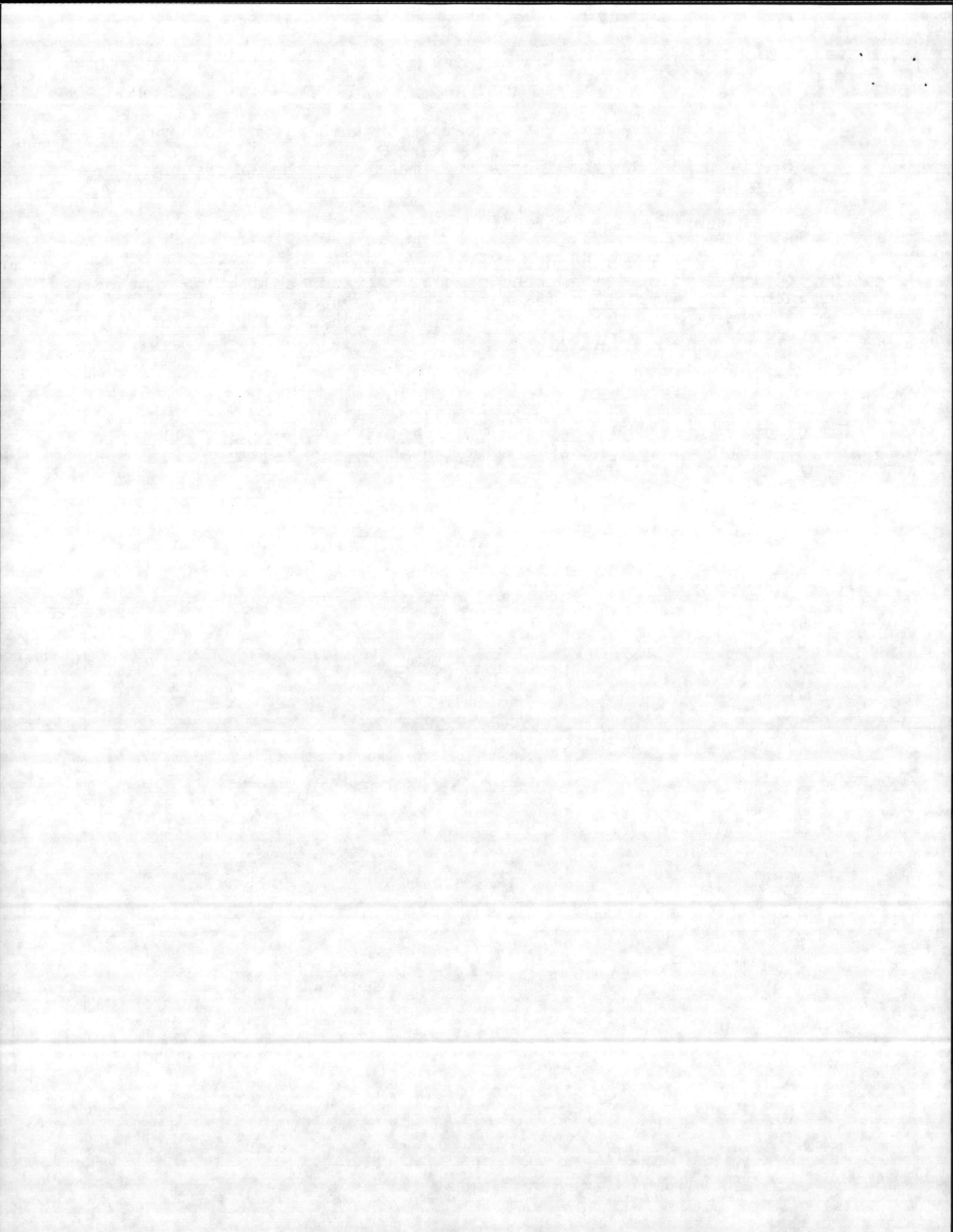
F. Upon leaving the laboratory, make sure the laboratory door is closed and locked.

G. Return the ice chests to plants.

III. Labelling - The laboratory will not use samples that do not have the initial sample time and the name of operator collecting samples for each shift samples collected. If an operator has collected a sample over two shifts, then he/she should put a (2) by his/her name.

Note 1

For plants manned 24 hr/day sampling period is 24 hours. All other plants will be-sampled over a representative eight hour period.



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SAMPLE SIZE CHART

Composite sampling information for #1,2 and 3 samples gathered for BOD₅ and suspended solids analysis. (See note 1)

- A. PLANT: Camp Johnson
- B. SAMPLING FREQUENCY: Tuesday, Wednesday, Thursday and Friday
- C. SAMPLING PERIOD: 0800-1600 hours
- D. SAMPLING INTERVAL: Once every hour
- E. FLOWMETER: Use meter located at Chlorine contact chamber to determine bottom scale reading required below.
- F. SAMPLE SIZE CHART:

Bottom Scale Reading

Sample Volume

0.0 - 0.5	125 ml
0.5 - 1.0	250 ml
1.0 - 1.5	375 ml
1.5 - 2.0	500 ml

Note 1

Please contact your supervisor/leader immediately for guidance if problem encountered relative to using flow meter to determine size of hourly sample. Each hourly sample should be promptly poured into appropriate one gallon sample container which is stored in refrigerator at plant. Do not worry if gallon jar is not full at end of sampling period. Actual volume collected will vary from day to day.

Grab Sampling Procedure-Wastewater Treatment

I. Tests

1. Total Residual Chlorine
2. pH
3. Dissolved Oxygen

II. Location: Sample Point 3 (III), which is at the end of the chlorine contact chamber.

III. Apparatus

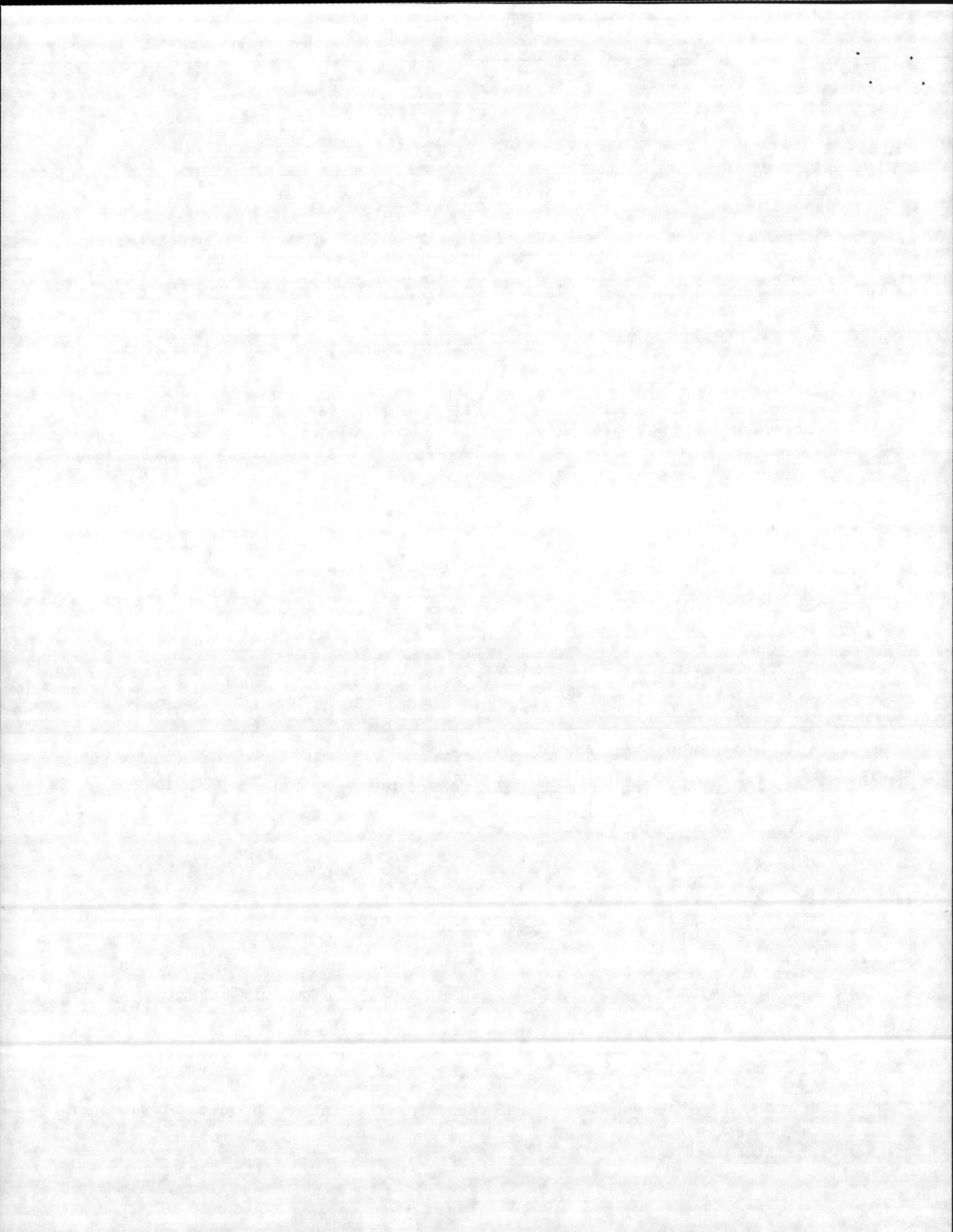
1. Plastic dipper (Note: No metal containers will be used)
2. Siphon

IV. Sampling Steps

1. Clean out sampler (dipper), if necessary.
2. Rinse the dipper 3 times with the effluent.
3. After the 3rd rinse, fill the dipper with the effluent.
4. Run the test immediately. Note: Use the siphon to fill the BOD bottle.
5. Rinse out the dipper with tap water.

V. The total residual chlorine and pH of the effluent taken by the Wastewater Treatment Plant Operators is reported to EPA and the State of North Carolina in the Quarterly Report.

Therefore to assure accurate readings the above procedure will be followed.



I. Sampling

1. Collect a representative sample in a BOD bottle, making sure no air is trapped.
2. Begin the D. O. test as soon as possible after the sample is collected.

II. Equipment—Supplied by Utilities Branch, BMaintDiv

1. 10 ml automatic buret
2. Safety pipet bulb
3. Gloves
4. Safety goggles
5. Rubber apron

III. Equipment and Reagents—Supplied by Quality Control Lab, Envir. Br., NREADiv

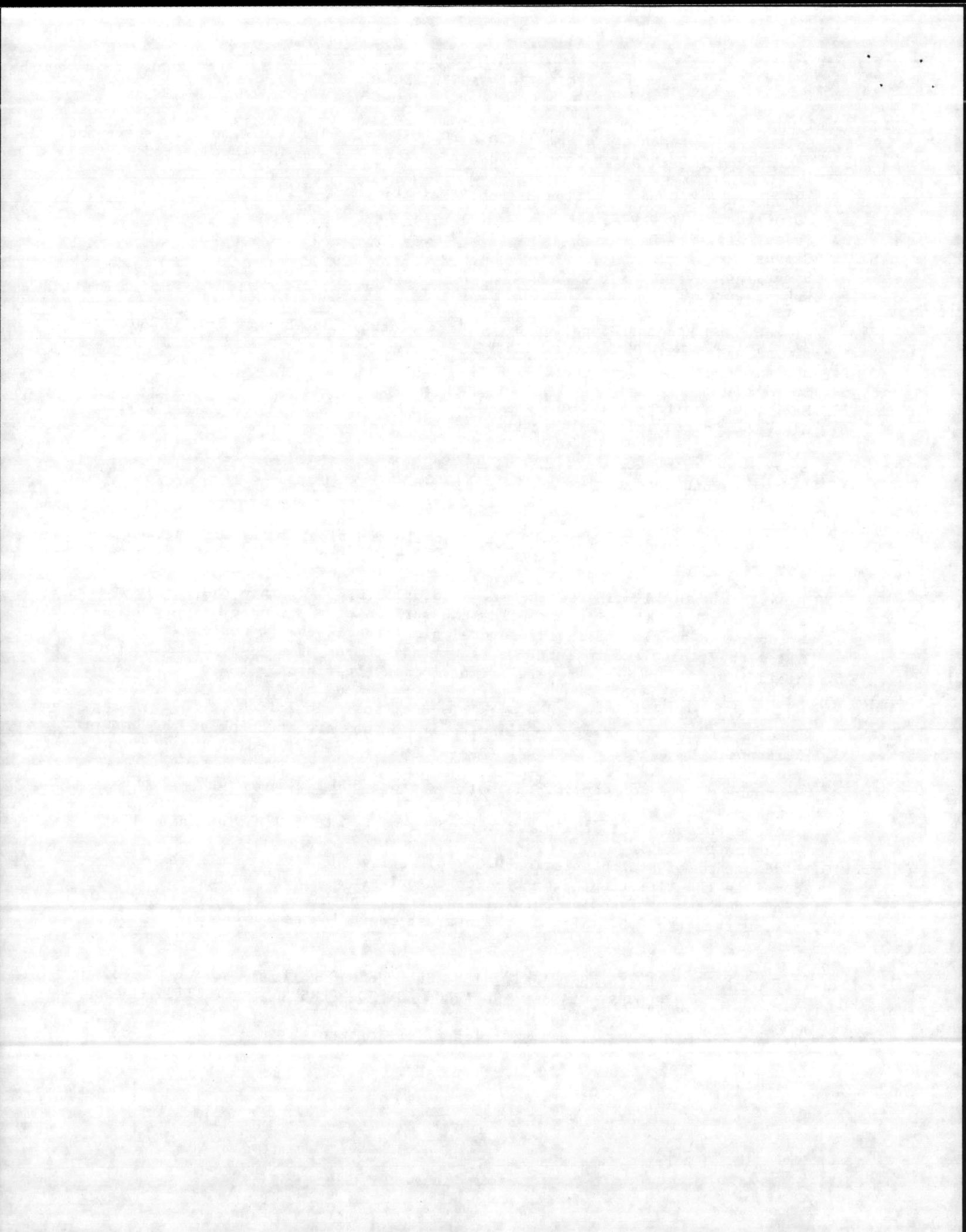
1. 500 ml wide mouth Erlenmeyer flask
2. 300 ml BOD bottle
3. 10 ml pipet
4. Manganese sulfate solution(D.O. #1)
5. Alkaline iodide azide solution(D.O. #2)
6. Concentrated Sulfuric Acid(D.O. #3)
7. Starch Solution(D.O. #4)
8. Sodium thiosulfate titrant, 0.0375 N(D.O. #5)

IV. Procedure

1. Remove the stopper from the full BOD bottle and pipet 2 mls of manganese sulfate solution(D.O. #1) into the sample making sure the tip of the pipet is below the surface of the sample.
2. Pipet 2 mls of alkaline iodide azide solution(D.O. #2) into the sample making sure the tip of the pipet is below the surface of the sample.
3. Stopper the bottle and invert several times to mix the flocculent.
4. Allow the flocculent to settle at least two thirds of the way down the bottle.
5. Again invert the bottle several times to mix the flocculent and allow to settle as in step 4.
6. Unstopper the bottle and add 2 mls of conc. sulfuric acid(D.O. #3) to the sample. Add the acid slowly allowing it to run down the neck of the bottle.
7. Restopper the bottle and invert rapidly several times to dissolve the flocculent.
8. Pour the entire contents of the bottle into a 500 ml wide mouth Erlenmeyer flask.
9. Titrate the sample to a light straw color with 0.0375 N sodium thiosulfate solution(D.O. #5).
10. Add one ml of starch solution(D.O. #4), swirl the flask to mix the sample, complete the titration, from blue to colorless.
11. Record the mls of sodium thiosulfate used. In this case, 1 ml=1 mg/l D.O.
12. Write the mg/l D.O. value to the nearest tenth in the log.

V. Comments

1. SAFETY NOTE: ALWAYS USE SAFETY PIPET BULB WHEN PIPETTING.
GLOVES, GLASSES AND APRON ARE FOR PERSONAL PROTECTION AND ARE TO BE WORN.
2. If any questions arise, see your leader or foreman.



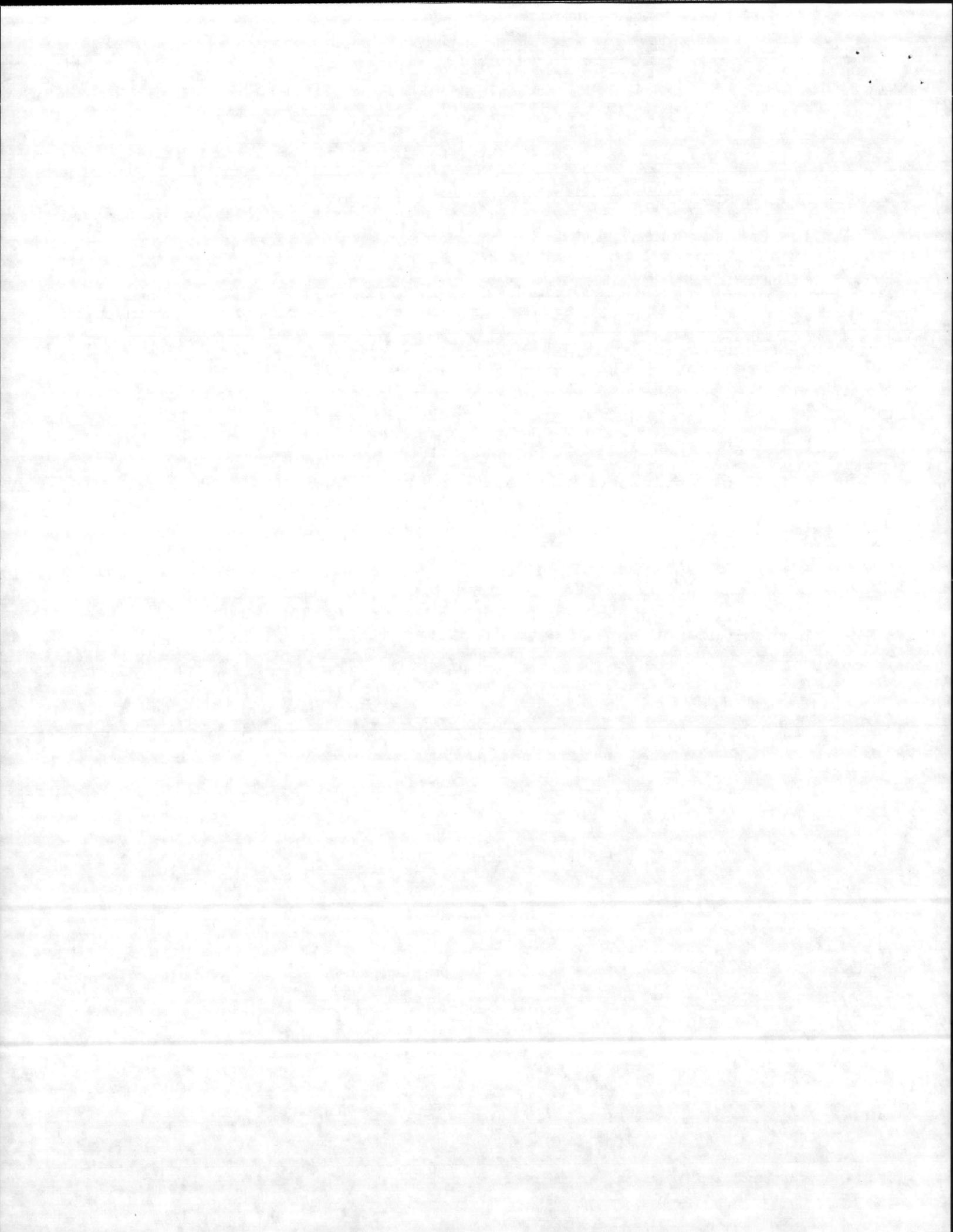
SETTLEABLE SOLIDS-WASTEWATER TREATMENT

I. Equipment-Supplied by Utilities Branch, BMaintDiv

1. Imhoff Cone
2. Stirring Rod

II. Procedure

1. Fill Imhoff cone to the liter mark with a thoroughly mixed sample.
2. Allow to settle for 45 minutes.
3. Gently stir the sides of the cone with stirring rod.
4. Allow to settle 15 minutes.
5. Record volume of settled matter in the cone as milliliters per liter (ml/l)
6. Where a separation of settleable and floating materials occurs, do not estimate the floating material.



Sampling Schedule-Wastewater Treatment

I. Plant Samples-Collected and Analyzed by Operators

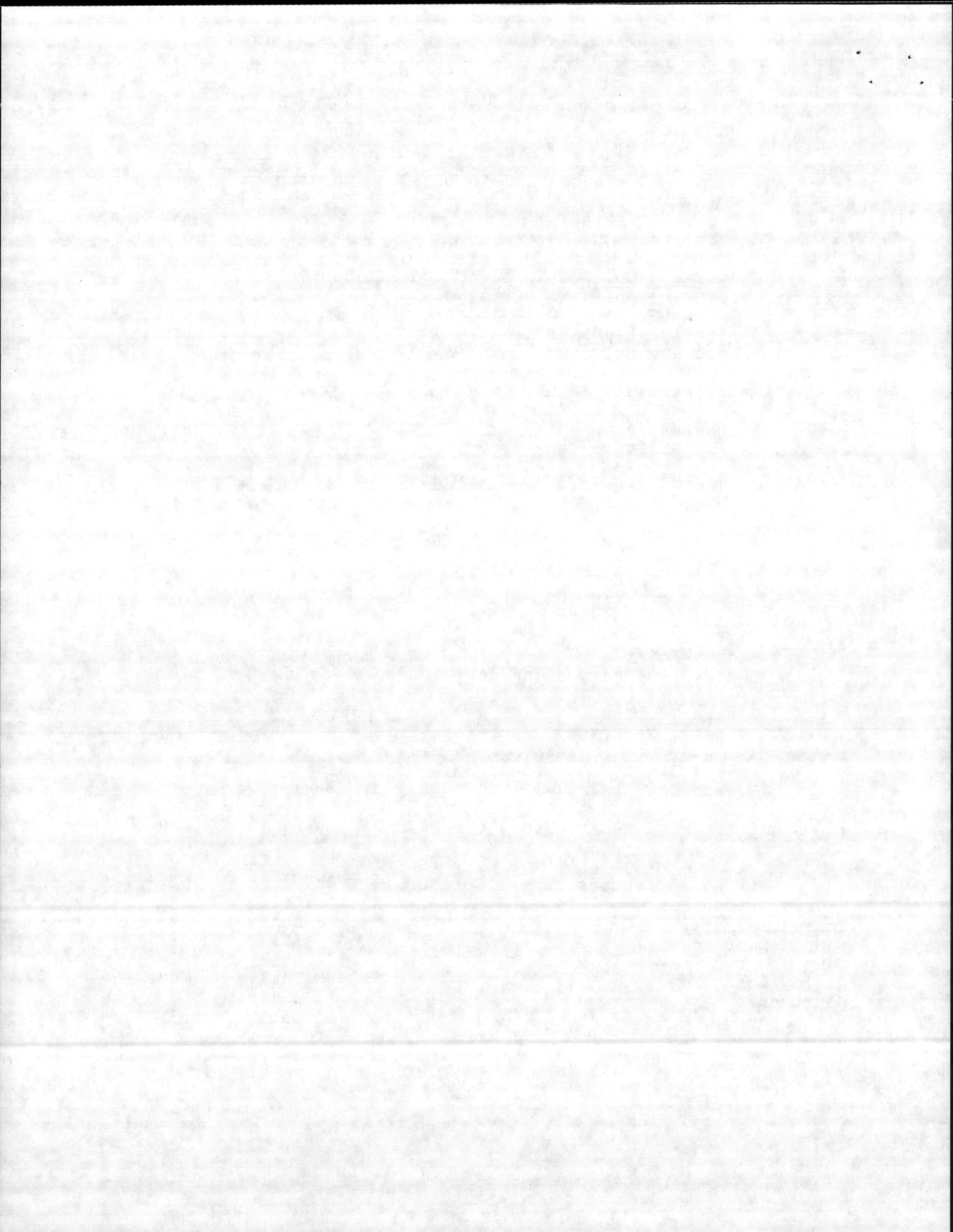
1. Dissolved Oxygen
8-4 Shifts: Once every Day
2. Settleable Solids (Influent and Effluent)
Once every Shift when manned
3. pH (Influent and Effluent)
Once every Shift when manned
4. Total Residual Chlorine
 - @ Hadnot Point
All Shifts: Once every hour
 - @ Tarawa Terrace, Camp Geiger
All Shifts: Once every hour
 - @ Camp Johnson, Onslow Beach, Courthouse Bay, Rifle Range
8-4 Shifts: Once every hour
12-8, 4-12 Shifts: Once a Shift

II. Coliform and Composite Samples

1. Hadnot Point
Monday ~~Sunday~~, Tuesday, Wednesday, Thursday, Friday
2. Tarawa Terrace, Camp Geiger, ~~Camp Johnson~~
Monday Tuesday, Wednesday, Thursday, Friday
Camp Johnson - Monday - Friday
3. Onslow Beach, Courthouse Bay, Rifle Range
Tuesday, Thursday

III. Required Sampling Frequency by NPDES Permit-Expires 26 Mar 1985

	Per Week			
	Composite	Coliform	Chlorine	pH
Hadnot Point	5	3	Daily	3
Tarawa Terrace	2	2	Daily	2
Camp Geiger	2	2	Daily	2
Camp Johnson	2	2	Daily	2
Onslow Beach	1	1	Daily	1
Courthouse Bay	1	1	Daily	1
Rifle Range	1	1	Daily	1



Supplies Obtained from Water Quality Control Laboratory

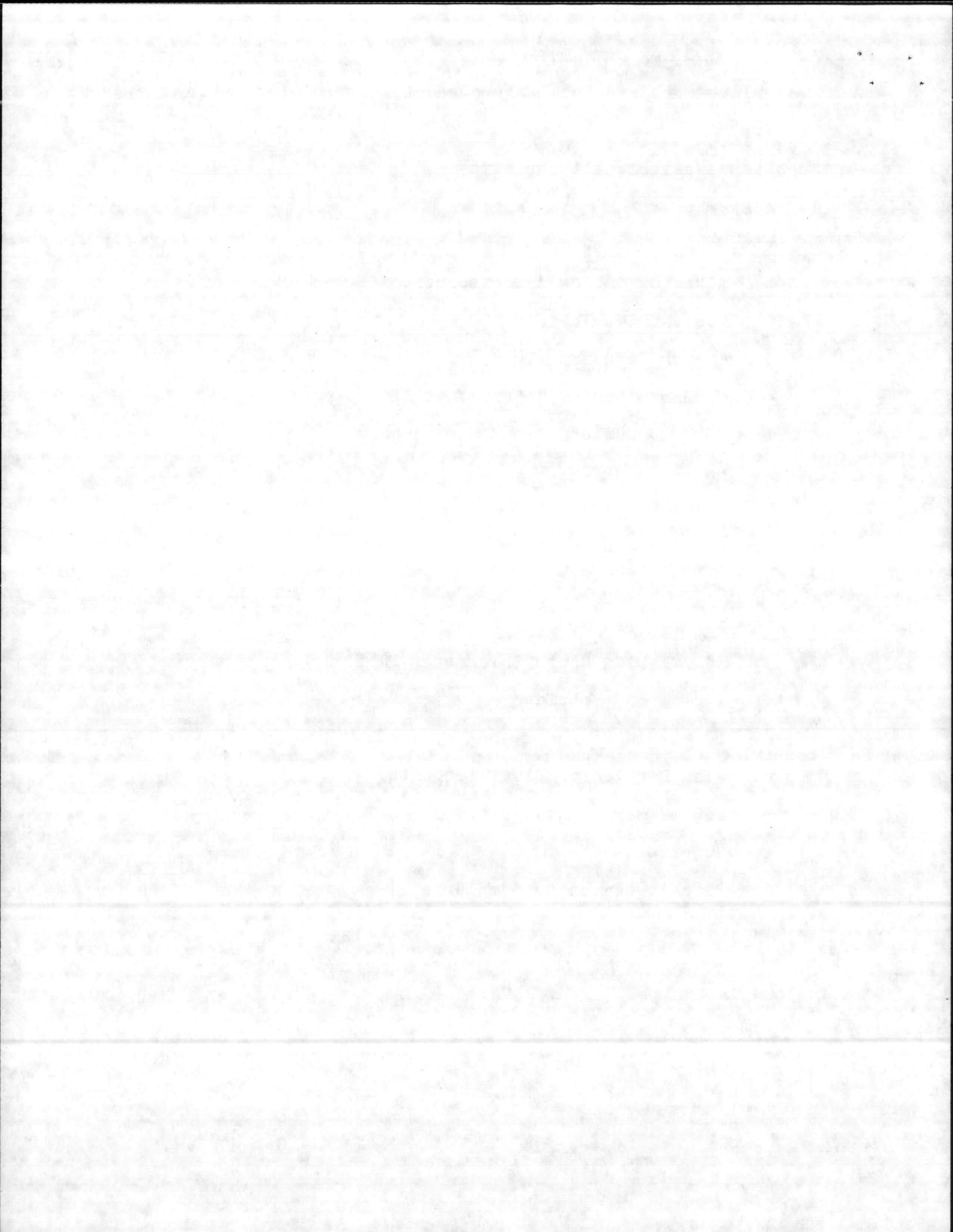
A. Supplies available all the time

I. Reagents

- a. Manganese sulfate solution (DO 1)
- b. Alkaline iodide azide solution (DO 2)
- c. Conc. Sulfuric Acid (DO 3)
- d. Starch Solution (DO 4)
- e. 0.0375 N Sodium Thiosulfate (DO 5)
- f. pH 7.00 Buffer
- g. pH 4.01 Buffer
- h. pH 9.18 Buffer
- i. Distilled Water
- j. Ammonia

II. Equipment

- a. 500 ml Wide Mouth Erlenmeyer Flask
- b. 300 ml BOD Bottle
- c. 10 ml Pipet
- d. Wash Bottle for Distilled Water
- e. 150 ml Sample Beaker (Plastic)
- f. Stirring Rods
- g. Plastic Composite Samplers
- h. Label Pens



B. Supplies available only by request from Foreman or Leader

I. Reagents

- a. For Amperometric Titrator Method
 - 1. pH 4 Buffer
 - 2. 5% KI solution
 - 3. Phenylarsine Oxide (PAO) Titrant
- b. For Ortho-tolidine Method
 - 1. Ortho-tolidine Chlorine Disc
 - 2. Ortho-tolidine Reagent

II. Equipment

- a. For Amperometric Method
 - 1. Amperometric Titrator
 - 2. 200 ml sample cup
 - 3. Glass Titrator
- b. For Ortho-tolidine Method
 - 1. Ortho-tolidine Disc

