PROCEDURES I FIELD COPY

WATER QUALITY CONTROL LABORATORY
NATURAL RESOURCES AND ENVIRONMENTAL AFFAIRS DIVISION
MARINE CORPS BASE
CAMP LEJEUNE, NORTH CAROLINA
LOCATED IN BLDG 65

LABORATORY, TREATMENT PLANTS AND FIELD TEST AND SAMPLING PROCEDURES NOTEBOOK

SEPTEMBER 1985

DISTRIBUTION

a. Master Copies (2)
   1. Contains All Sections
   2. Located
      A. Supervisory Chemist's Office (Rm 215-A)
      B. Technician's Office (Rm 216)

b. Water Laboratory Copy
   1. Contains Sections
      A. General Information
      B. General Laboratory Test Procedures
      C. Water Analysis Procedures Subsections
         1. Titrations (Wet Chemistry)
         2. Colorimetric Procedures (HACH Spectrophotometer
         3. Electrode Probe Procedures (Fisher Ion-Meter)
   2. Located in Water Laboratory (Rm 219)

c. Atomic Absorbtion Procedures Copy
   1. Contains Atomic Absorbtion Procedures Section
   2. Located in Water Laboratory (Rm 219)

d. Gas Chromatograph Procedures Copy
   1. Contains Gas Chromatograph Procedures Section
   2. Located in Water Laboratory (Rm 219)

e. Wastewater Laboratory Copy
   1. Contains Sections
      A. General Information
      B. General Laboratory Test Procedures
      D. Wastewater Analysis Procedures
      E. Treatment Plant Operator Procedures
   2. Located in Wastewater Laboratory (Rm 220)

→ f. Field Copy
   1. Contains Sections
      A. General Information Subsections
         1. General
         2. Safety
         4. Sampling
         6. Equipment Operation and Maintenance (Partial)
      E. Treatment Plant Operator Procedures
   2. Located in Technician's Office (Rm 216) or in Truck

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A General Info

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GENERAL INFORMATION

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SAMPLING SCHEDULE - WATER TREATMENT

I. Plant Samples - Collected and Analyzed By Operators

1. Alkalinity
   Once every two hours/filter (17)

2. pH
   By Meter
   @HP, HB, TT, AS - once every shift
   By comparator
   @ Pools - twice on 8-4, once on 4-12

3. Chlorides
   @RR, CHB, OB, CJ, AS (for wells) once/shift

4. Hardness
   Twice every two hours (treated & delivered)

5. Stability
   @ AS - once/shift
   @ HP - once/shift

6. Fluorides
   @ HB, HP, TT - once/shift

7. Chlorine Residual (?)
   Every two hours/plant

II. Weekly Sampling - Usually Tuesday

1. One Chemical with Chlorine Residual Per Plant

2. Bacteria Sampling

   A. HP - 6 Fixed Points; 3 Random Points
      HB - 4 Fixed Points; 3 Random Points
      } Sample #29-46
      SH-8
      Pools: Area 5
             Area 2
             P.P. O'Club
             P.P. O'Club Baby
      } when open

   B. TT - 2 Fixed Points; 1 Random Point
      CJ - 1 Fixed Point; 2 Random Points
      AS - 4 Fixed Points; 3 Random Points
      } Sample #13-28
      Pools: Camp Johnson
             Terawa Terrace
             MCAS E Club
             MCAS O' Club
             MCAS O' Club Baby
      } when open

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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 (24 Hr)
   Sunday, Tuesday, Wednesday, Thursday, Friday

2. Tarawa Terrace (24 Hr); Camp Geiger (24 Hr); Camp Johnson (8 Hr)
   Tuesday, Wednesday, Thursday, Friday

3. Onslow Beach, Courthouse Bay, Rifle Range (all 8 Hr)
   Tuesday, Thursday

III. Required sampling frequency by NPDES Permit - Expires 26 March 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

C. RR - 2 Fixed Points; 1 Random Point
CHB - 3 Fixed Points; 1 Random Point
OB - 1 Fixed Point; 1 Random Point
} Sample #1-12
BB-97
OB Pond pH & Solids Bottle

III. Require Number of Bacteria Samples Per Month By SDWA

HP	30	CJ	2
MCAS	13	RR	2
HB	9	CHB	2
TT	6	OB	1

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IN-LAB SAMPLE CHECKLIST

WATER SAMPLES

I. Preparing sample containers:

A. Chemical Analysis 1 Liter Plastic Bottle

1. Wash bottles with soap and water or run through dishwasher.

2. Rinse bottles with distilled water, can use distilled water rinse cycle on the dishwasher.

3. Place label tape (white) on bottle as sample bottles are released.

B. Coliform Bottles

1. Wash bottles in dishwasher with distilled water rinse.

2. Add Sodium Thiosulfate.

3. Put autoclave indicator tape on lid and autoclave bottle and lid w/sodium thisulfate inside.

4. Run quality control check for sterility on one bottle in batch.

5. Place label tape (white) on bottle side as bottles are released (if needed).

II. Receiving Sample containers:

A. Labels

1. Check labels for the required information

a. Chemical

(i) Bldg. No.
(ii) Chlorine residual

b. Coliform Bottles (information may be on accompanying form)

(i) Date
(ii) Time
(iii) Chlorine residual
(iv) Name of collector
(v) Names of anyone else who had custody of sample before turned over to Lab (if needed)

B. Screen samples for Chlorine

C. Process samples

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TABLE 5

DIRECTORY OF APPROVED WASTEWATER PLANT OPERATORS FOR SAMPLE COLLECTION

ALDRIDGE, Barry T.
AMBROSE, John H.
ANTINORI, David L.
BROWN, Clennie L.
CARLYLE, Billy B.
COLLINS, Edward G.
~~CONNOR, Joe A~~.
CREWS, Stephen V.
~~DARDEN, Glenn L~~.
DAVILA, Gabriel
DAVIS, Mack D. Jr.
DELGADO-NIEVES, Dolores
FARLAND, Melvin S.
FARROW, McArthur
FUTREAL, Rupert
FUTRELL, Norvin J.
GODWIN, Otis E.
HALL, Leitha W.
HILL, Stanley E.
HUDGINS, Alton O.
KELLUM, Kenneth D.
KENNEDY, Tommie H.
NORRIS, Rebecca E.
PACK, Donald L.
PATE, James C.
RHODES, Randal B.
ROLLINGER, David L.
SARDINAS, Frank
SAULTER, Albert F. Jr.
~~SCHMIDT, Carroll V~~.
SNODGRASS, Anthony P.
SNODGRASS, Pamela C.
STEVENSON, David M.
TAYLOR, Herman B.
TAYLOR, Johnnie P.
THOMPSON, James L.
TREDWELL, David H.
WILLIAMS, Victor W.
WOOLDRIDGE, Earl C.
YOPP, Everett D.

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RIVER WATER PROCEDURES

The river is sampled once a month.

I. Sample Collection Points

1. RW-1 - Hughes Marina
   RW-2 - Sight Bridge (NC-24)
   RW-3 - Knox Trailer Park
   RW-4 - Hospital Point
   RW-5 - Marker #35
   RW-6 - Sneads Ferry Bridge
   RW-7 - Marker #12
   RW-9 - Marker #65A
   RW-8 - Onslow Beach Bridge

2. Sample Point Collection Relative to Outfall of Plants

Plant	Upstream	Downstream
C.G.	RW-01	RW-04
T.T.	RW-02	RW-03
M.P.	RW-03	RW-04
H.P.	RW-04	RW-05
R.R.	RW-05	RW-06
CHB	RW-06	RW-07
OB	RW-08	RW-09

3. Type of Samples Collected Each Sample Site (RW-1 - RW-09)

   (A) Two (2) BOD bottles for DO and BOD_5 determination
   (B) One (1) grab sample for fecal, total coliform and pH determination
   (C) One (1) grab sample for oil and grease
   (D) One (1) grab sample for bacti from each outfall
   (E) Six (6) grab samples for bacti from Wallace Creek during summer months.
   (F) Water Temperature
   (G) Tide Condition
   (H) Time of Sampling

II. Boat Procedure

A. Before leaving laboratory check and be sure all sample bottles are ready (Table 1). Get life preservers, fire extinguisher, anchor, ice chest, gas card and clip board.
B. Get radio from NREAD (See radio procedure)
C. River Water Sampling
   1. Log sample time .
   2. Upon arrival at sampling point, the BOD bottles are placed in apha type sampler and submerged to fill.
   3. A grab sample for bacti and pH is collected.
   4. A grab sample for oil and grease is collected.
   5. Water temperature is taken and recorded.
   6. Time recorded

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Collection

Procedures A-19

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COURTHOUSE BAY PROCEDURES

I. Courthouse Bay will be sampled twice a month. Preferably once before rain and once after. This has been requested by the AC/S Facilities to gather some background data related to putting in a new ramp for Amtracs.

II. Sample Collection Points

- A. 9 points
- B. 3 from the boat
- C. 6 from shore
- D. Locations and Numbers - see map
  1. Points 1 & 2 are on either side of ramp in water
  2. Point 3 is mouth of bay
  3. Point 4 is marsh, far side of ramp
  4. Point 4A - man-made ditch, runoff from construction above A-3
  5. Points 5, 6 and 7 - new storm drains
  6. Point 7A - old storm drain

III. Sample Collection - All Points

- A. 2 BOD Bottles (treat one with DO1 & 2)
- B. Oil and Grease
- C. Bact Bottle w/o Sodium Thio

IV. Sample Analysis (Laboratory)

- A. Dissolved Oxygen
- B. BOD 5-Day
- C. pH
- D. Oil and Grease
- E. Total Coliform
- F. Fecal Coliform
- G. Fecal Strep

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IN-LAB SAMPLE CHECKLIST

WASTEWATER SAMPLES

I. Preparing Sample Containers:

A. Composite jars

1. Wash jars with soap and water or run through dishwasher
2. Rinse jars with distilled water, can use distilled water rinse cycle on the dishwasher.
3. Put a permanent label on jar and lid - that is a white label with plant and sample number with clear tape over it - if needed.
4. Place label tape (white) on jar lids as sample jars are released.
5. Replace jar lids as they rust.

B. Coliform bottles

1. Wash bottles in dishwasher with distilled water rinse.
2. Put a permanent label on bottle, if needed - that is a white label with the plant with clear tape over it.
3. Add Sodium Thiosulfate.
4. Put autoclave indicator tape on lid and autoclave bottle and lid w/sodium thio inside.
5. Place label tape (white) on bottle side as bottles are released.

II. Receiving Sample Containers:

A. Labels

1. Check labels for the required information

a. Composite jars

(i) Initial time
(ii) Operator Name(s), 3 for HP, TT & CG, 1 for CJ, OB, CHB, RR

b. Coliform Bottles

(i) Date
(ii) Time
(iii) Chlorine Residual
(iv) Name (only one)

2. Compare names on labels with the posted list of approved operators (if name is not approved or illegible, circle in red and report it to the Supervisory Chemist)

3. Mark labels with plant and sample

4. Pull label off and place on back of lab worksheet

B. Screen samples for Chlorine - Indicate chlorine found on lab sheet & sample label

C. Process samples

IV. Wallace Creek Sample Locations:

A. #1 Entrance to Marina.
B. #2 Enlisted Pier.
C. #3 Under power line, middle of bridge.
D. #4 Left bank, going up stream, at first ditch. (Nest on top of dead tree).
E. #5 Two pines leaning over water, one on each bank.
F. #6 Second Bridge, back gate road.

7. The apha sampler is recovered and the BOD bottles removed.
   a. One is tightly stoppered for BOD_5 incubation on return to laboratory.
   b. The other is preserved by the addition of manganous sulfate (2 ml) and alkaline iodine-azide (2 ml), stoppered and shaken 25 times. This bottle is for DO determination on return to laboratory.
8. Upon completion of trip, the boat will be gassed and washed prior to returning to laboratory.

D. Outfall Sampling
   1. Log sample time
   2. Grab coliform sample
   3. Take chlorine residual

E. Brynn Marr, Wilson Bay and Wallace Creek
   1. Log sample time
   2. Grab coliform sample

III. Laboratory Procedures

A. One BOD bottle to incubator for BOD_5 determination

B. The other bottle is used for DO determination, using the winkler full bottle procedure.

C. Set up fecal and total coliform cultures.
   1. DO positive and negative controls
   2. Set up two dilutions on each sample point. Usually the dilutions used will be 1 ml and 10 ml for total and 1 ml and 25 ml for fecal. These volumes may increase or decrease according to rain, etc. Generally, follow history of past results for site tested.
   3. Do pH on each sample
   4. Acidify the grab sample for oil and grease.
   5. The next day, perform the oil and grease analysis. Read coliform plates and record.
   6. After five (5) days incubation, determine BOD_5 on second BOD bottle and compute depletion as found under winkler methodology.
   7. Compile all results for monthly report.

TABLE
SAMPLE BOTTLES

# Bact Bottles Tottle	18 (summer months add 6 more)
# BOD Bottles .	18
# Oil and Grease	9

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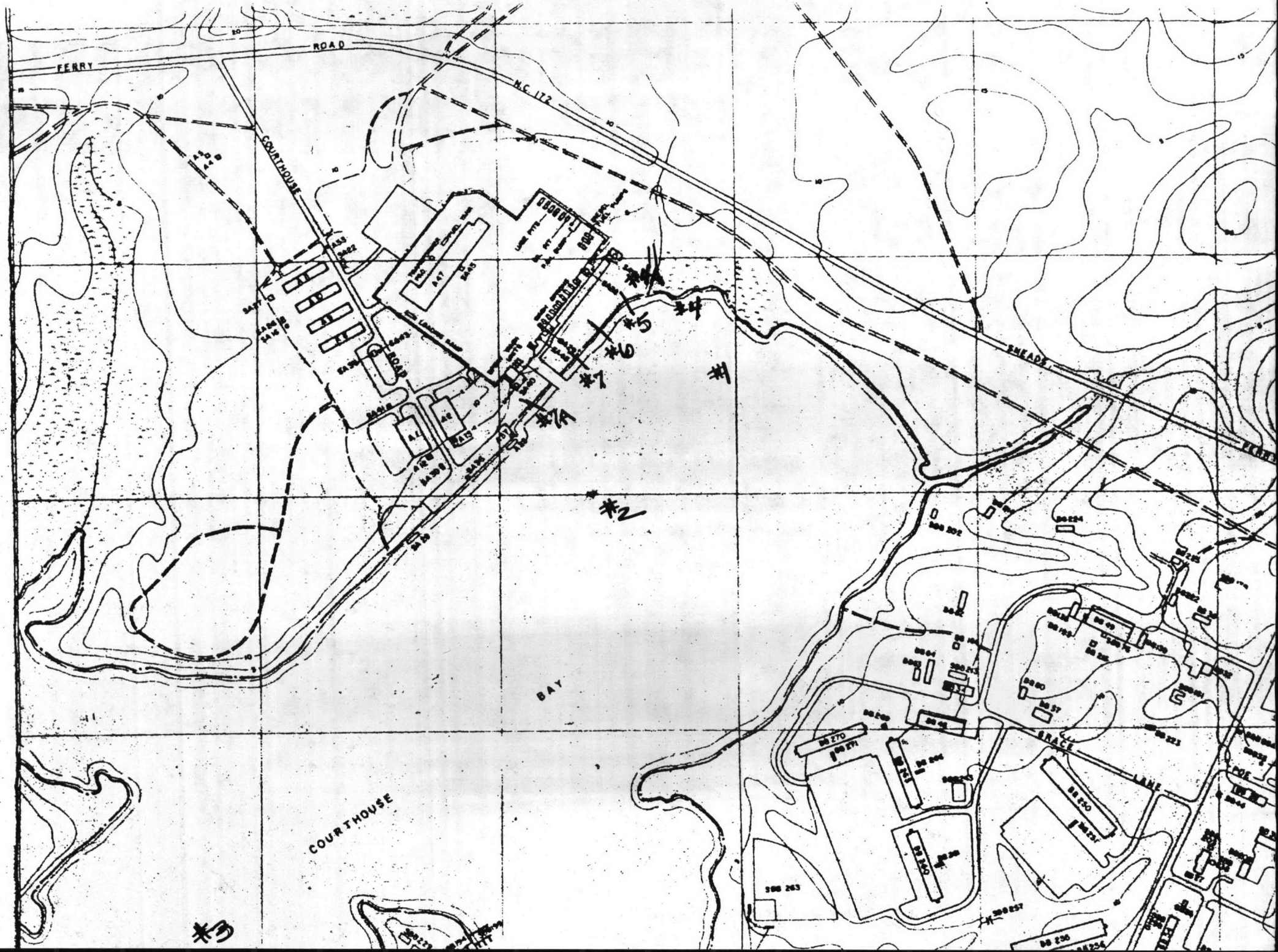
FERRY
ROAD
NC 172
COURTHOUSE
ROAD
SHEADS
BAY
COURTHOUSE
GRACE
LANE
#1
#2
#3
#4
#4A
#5
#6
#7
#7A

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STORM DRAIN SAMPLE COLLECTING PROCEDURES

I. FREQUENCY:
Each storm drain is sampled 4 times a year. Samples are usually taken in sets of eight from geographically related areas of the Camp Lejeune Complex. The number of storm drains requires a sampling run usually once a week.

II. SAMPLE TYPES (same at each location):
Oil and Grease--A 1.0 liter sample in wide-mouth glass bottle with ground glass top.
Suspended Solids and pH--Approximately one liter sample in Nalgene polybottle. Do not delay taking samples to laboratory for accurate pH determination.

III. COLLECTION PROCEDURE:
Suspended Solids and pH--Colèect this first, so silt will not be disturbed. Carefully submerge bottle in stream, with neck upstream. If water is silty, a full liter of sample won't be necessary.

Oil and Grease--Rinse bottle once with water from the sample site. Refill to halfway up the curve on the shoulder of the bottle. (About an inch and a half from the top.)

IV. STREAMFLOW ESTIMATION:
A) Flowing Streams--
1. Find a stretch of the stream where the flow is straight and smooth.
2. Measure the width and average depth in feet.
3. Determine the flow velocity in feet per second--
   a) Mark off a two or three foot section of stream.
   b) toss in a bit of debris (Witch hazel seed pods work very nicely for this. They're round and dense, but still float.), and time its passage .
   c) Divide the number of feet in the marked section by the number of seconds to determine feet per second flow.
   d) Try a couple of times and determine the average.
4. Calculate flow in gallons per day--
   a) Multiply width X depth X flow in feet per second to give cubic feet per second (cfs).
   b) Multiply by 584,200 to give gallons per day.

B) Non-flowing Storm Drains--
Log as either dry or no flow, whichever is more accurate.

V. PRESERVATION:
The oil and grease sample must be preserved by the addition of five milliliters of 1:1 HCl or H_2SO_4 to the sample as soon as possible after returning to the laboratory. Shake well to mix.

VI. ANALYSIS:
A) pH is determined immediately upon return to the laboratory.
B) TSS is determined the same as for sewage.
C) Oil and Grease analysis must be done within 24 hours.
The analysis method is outlined in Standard Methods.
Run a blank and a standard, as well.

STORM DRAIN SAMPLE COLLECTING PROCEDURES

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Suspended Solids and pH--Approximately one liter sample in Nalgene polybottle. Do not delay taking samples to laboratory for accurate pH determination.

III. COLLECTION PROCEDURE:

Suspended Solids and pH--Collect this first, so silt will not be disturbed. Carefully submerge bottle in stream, with neck upstream. If water is silty, a full liter of sample won't be necessary.

Oil and Grease--Rinse bottle once with water from the sample site. Refill to halfway up the curve on the shoulder of the bottle. (About an inch and a half from the top.)

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   d) Try a couple of times and determine the average.
4. Calculate flow in gallons per day--
   a) Multiply width X depth X flow in feet per second to give cubic feet per second (cfs).
   b) Multiply by 584,200 to give gallons per day.

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VII. REPORTS:
Findings are reported to the Supervisory Chemist, and (eventually) to EPA. If visible oil, extensive suspended solids, or the like is noted when you make your sample visit, make a quick determination of the source, if possible, and advise the Supervisory Chemist for possible further investigation.

VIII. Revised By: Elizabeth A. Betz
Date: 22 August 1985

SNEADS FERRY ROAD LANDFILL SAMPLE COLLECTION PROCEDURE

I. Frequency: Once a week, as directed.

II. Sample Sites:

C-1 Cogdel's Creek at crossing of the Sneads Ferry Road.
C-2 Headwaters of Cogdel's Creek in the swamp upstream of the landfill.
CH-1 Cowhead Creek at crossing of Sneads Ferry Road.

III. Sample Types (same at all locations):

A) Two 300 ml BOD bottles for DO and BOD determinations
B) One large bac-t sample bottle (without thiosulfate) for total and fecal coliform determination.

IV. Equipment:
Sample bottle carrier, Clark D.O. sampler, and insect repellant.

V. Sample Procedure:

A) Take the coliform sample first. Avoid touching the threads on the top or contaminating the inside of the bottle. Fill bottle to the neck, leaving about three-quarters inch of air space.

B) Load BOD bottle into sampler, affix cap, and immerse sampler in stream deep enough to cover (by ½ inch) the air outlet tube. Take care not to disturb stream bottom. When bubbling stops, retrieve sampler, and carefully replace the ground glass stopper, taking care not to introduce any air. Take the second BOD sample immediately and in the same spot as the first.

C) Transport samples immediately to the laboratory for accurate determinations of D.O. and pH.

VI. Reporting:
Laboratory findings are reported to the Supervisory Chemist and are kept in a bound notebook in the laboratory for possible future reporting.

VII. Revised by: Elizabeth A. Betz
Date: 22 August 1985

SNEADS FERRY ROAD LANDFILL SAMPLE COLLECTION PROCEDURE

I. Frequency: Once a week, as directed.

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Date: 22 August 1985

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VIII. Revised By: Elizabeth A. Betz
Date: 22 August 1985

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VOLATILE ORGANICS COLLECTION PROCEDURES

I. Trihalomethanes (THMs)

A. Use clean Supelco 40 ml sample vials

B. Add sodium thiosulfate crystals to vials

C. Collect in duplicate

D. Fill vials completely, screw cap on

E. Check for air bubbles by gently tapping cap against hand

F. If air bubbles are present, take cap off add more water. Gently repeat check until no air bubbles are present.

G. SDWA THMs must be collected for each system in the same day and at the same locations each time. See list below for locations.

II. All Volatile Organics (VOCs)

A. Same as THMs above, except omit steps B and G.

B. Locations will vary from any drinking water point, raw water wells or test wells.

III. Trihalomethane Locations Required by SDWA

A. New River Air Station Water System

1. New River Water Treatment Plant, Bldg. AS-110, at Pump

2. Career Planner, Bldg. G-520, Men's room sink, 2nd floor

3. Barracks Rec. Room, Bldg. 4025, Bathroom sink, 1st floor

4. Officer's Club, Bldg. 710, Gally Sink

5. Boat Marina, Bldg. 2800, Men's room sink

B. Hadnot Point Water System - See Note Below

1. Hadnot Point Water Treatment Plant, Bldg. 20, at Pump

2. Naval Hospital, Bldg. NH-1, Emergency Room Sink

3. Base Maintenance, Bldg. 1202, Men's room sink, 1st floor

4. Quality Control Lab, Bldg. 65, Room 220 sink

5. Barracks, Bldg. FC-530, Laundry Room Sink, 1st floor

C. Other Streams - See THM File 11333/1

NOTE: Hadnot Point is presently required to take only one sample at FC-530 because it has low THM levels.

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HYDRAZINE COLLECTION PROCEDURES

I. PURPOSE:

Not all boilers aboard Camp Lejeune have backflow preventors (check values). Until all check values are installed the Water Quality Control Laboratory will randomly sample drinking water for hydrazine.

II. PROCEDURE:

A. Run water for a few minutes.

B. Collect a minimum of 100 mls in a clean plastic or glass bottle.

C. Fill completely and cap tightly.

D. Avoid excessive agitation.

E. Analyze as soon as possible - there is no holding time.

Trace Metal Sampling

A. Sample Containers:
1. Plastic
2. One liter size

B. Cleaning Procedure:
1. Detergent Wash
2. Tap Water Rinse
3. Nitric Acid Rinse
4. Distilled Water Rinse, three times

C. Preservatives: Nitric Acid
1. To a pH less than 2.5
2. About 10 ml/liter

D. Label with:
1. Activity-Camp Lejeune Marine Corps Base (only if shipped out Base)
2. Sampling Point
3. Name of Collector
4. Date
5. Time
6. Treated w/Nitric Acid

E. Stor-age:
1. At room temperature
2. For 6 months

F. Revised by: Elizabeth A. Betz
Date: 22 August 1985

SEP 20 1985

HYDRAZINE COLLECTION PROCEDURES

I. PURPOSE:

Not all boilers aboard Camp Lejeune have backflow preventors (check values). Until all check values are installed the Water Quality Control Laboratory will randomly sample drinking water for hydrazine.

II. PROCEDURE:

A. Run water for a few minutes.

B. Collect a minimum of 100 mls in a clean plastic or glass bottle.

C. Fill completely and cap tightly.

D. Avoid excessive agitation.

E. Analyze as soon as possible - there is no holding time.

Trace Metal Sampling

A. Sample Containers:
   1. Plastic
   2. One liter size

B. Cleaning Procedure:
   1. Detergent Wash
   2. Tap Water Rinse
   3. Nitric Acid Rinse
   4. Distilled Water Rinse, three times

C. Preservatives: Nitric Acid
   1. To a pH less than 2.5
   2. About 10 ml/liter

D. Label with:
   1. Activity-Camp Lejeune Marine Corps Base (only if shipped out Base)
   2. Sampling Point
   3. Name of Collector
   4. Date
   5. Time
   6. Treated w/Nitric Acid

E. Stor-age:
   1. At room temperature
   2. For 6 months

F. Revised by: Elizabeth A. Betz
   Date: 22 August 1985

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Bacteriological Sampling Procedure

1. General.

a. The purpose of the sampling os to insure disinfection and that bacteria do not exceed the limits established by the Safe Drinking Water Act of four per 100 militers (mls), or more than an average of one per 100 mls for the distribution system.

b. It is extremely important that proper precautions and techniques are used to preclude water samples from becoming contaminated with bacteria from hands, clothing, etc.

c. DO NOT take samples form outside spigots, leaking spigots, swing spigots or from spigots where the sanitation is highly questionable.

d. When taking samples from spigots that have aerators, remove the aerator before running the water (to waste) and collecting the sample. After the sample is collected replace the aerator.

2. Apparatus.

a. Sterile sample bottle, approximately 100 mls, with label.

b. Chlorine Test Kit, if water is chlorinated.

c. Equipment to flame spigots(optional)
   1. Foreceps
   2. Jar containing alcohol-saturated cotton balls
   3. Lighter or matches

d. Iced coolers for storage(Required if sample can't be delivered to Quality Control Lab within 1 hour).

3. Sampling Procedure.

a. Select a proper sample point.

b. Label sample bottle with sample point identification(ie. Bldg #, Unit, Facility, Area).

c. Remove faucet aerator, if necessary, and run the water (to waste) for five minutes.

d. Perform a chlorine check on the water and record the results on the label, note time, date and collector on label. (Note: Chlorine residual should be 0.2 mg/l, or higher).

e. Shut off the water and flame the spigot for about one minute, if desired. Turn water on again and run (to waste) a few more seconds.

f. Remove the top of the sample bottle, taking care not to handle the neck of the bottle or the inside of the cap, and collect about 100 mls of sample. (Note; DO NOT rinse the bottle. DO fill only to the shoulder of the bottle, leaving about one inch of air space).

g. Recap the bottle and return it to the sample carrier.

h. If not possible to get to Lab within 1 hr place in a iced cooler.

i. Return the samples to a technician in the Lab by 1400(Monday-Fridays)

j. The maximum holding time for the sample, from collection to analysis, is 30 hours. Sampling and transporation should be arranged, keeping in mind the 30 hr holding time and the 1400 deadline. The laboratory normally can not set up samples on Saturday unless the reason is sufficient to justify overtime.

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MODEL 1680 ISCO AUTO SAMPLER

I. Power Source:
   A. Nicad Battery Packs
      1. Connect Nicad Battery to ISCO Sampler at power outlet. (Charge for 24 hours before using)
      2. Use AC/DC power pack charger when possible.

II. Setting Up Sampler's Location
   A. Locate at sewage plant the I, II, and III locations.
   B. Unroll intake tubing from sampler.
   C. Place strainer in water (make sure that strainer is not located against wall or bottom of tank).
   D. Place ice in bottle tube for cooling purpose.

III. Setting Up Sampling Program
   A. Set Mode Switch on TIME
   B. Set Sample Internal Switch at (amounts of minutes when you want first sample taken. Time could vary from 1 to 999 minutes.)
   C. Set Multiplexer on "Bottle per Sample"
   D. Set Suction Line Length at 13 1/3, 29 1/3 setting
   E. Set Sample Switch to 1
   F. Set Pump Switch to AUTO
   G. Press Set Delay button
   H. Press Display button (make sure 060 is on display)
   I. Press Manual Advance until part shows bottle #1
   J. Set Volume Selector 390-132
   K. Set Sample Internal Switch at 060. (Do not press delay button.)
   L. Sample program is complete and ready.

IV. Sample Handling
   A. After cutting sampler off, remove top and expose bottle tube.
   B. Cap all bottles off for transportation to lab.
   C. At lab, pour sample from each tube I, II, III into large container for composite sample.
   D. Pour off one gallon for analysis.

NOTE: For future reference and explanation, please refer to Model 1680 Instruction Manual. For volume selection, always check volume before leaving sampler for night.

V. Revised by: Gaines B. Huneycutt
   Date: 2 October 1985

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MODEL 1640 - LIQUID LEVEL SAMPLER ACTUATOR SOP

I. Description - The Model 1640 is a device used in conjunction with an ISCO Wastewater Sampler to begin a sampling routine when a liquid level reaches same predetermined height.

A. Components
   1. Control Box
   2. 22 ft. Coaxial Cable
   3. Probe Assembly and Clamps

B. ISCO Wastewater Samplers Compatable with the 1640
   1. All late model ISCO samplers (Model 1680, Water Quality lab samplers)

II. Preparation for Use

A. Connection to an ISCO Wastewater Sampler
   1. Place the Model 1640 on top of sampler's battery pack.
   2. Set the sampler's Pump switch to OFF.
   3. Program the sampler to take samples at the desired time intervals. (To be determined per location used)
   4. Set sampler in Time mode.
   5. Set Control switch on Model 1640 to the Toggle/Reset position to reset actuator.
   6. Connect sampler connector to the Flowmeter socket (beside power supply connector).

III. Mounting Probe Assembly

A. Place Model 1640 Probe assembly rigidly above the flow stream using weather resistant hardware. (Probe must be within 22 ft. of sampler.)

B. Actuation will occur when liquid level reaches the starters steel ring inside rain cap.

IV. Latch Mode - While operating in the LATCH mode, the 1640 will actuate the sampler when the level rises to the stainless steel ring on the probe. The sampler then remains actuated even if the liquid level recedes and no longer touches the probe. When first actuated, the sampler will immediately take a sample. The sampler will return to its prescribed time base and take samples at the programmed time intervals.
Example: The Model 1640 is set to the Latch Mode and the sampler is turned on at 4:00. The sampler is programmed to take a sample every hour on the hour. At 6:20 (more than one time interval between samples has passed) the liquid level rises to the probe, and the 1640 actuates the sampler. The sampler then takes the first sample at 6:20, the second at 7:00, the third at 8:00, etc. and continues taking samples every hour until it completes its entire program. However, if the sampler is turned on at 4:00 and the liquid level rises to the probe at 4:20 (less than one time interval between samples has passed), then the sampler will take the first sample at 5:00, the second at 6:00, the third at 7:00, etc. until it has completed its entire program.

A. Programming the Latch Mode (assuming that steps II, A, 1-6 have been completed)

   1. Place sampler pump switch in the AUTO position.

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2. Momentarily set Model 1640 Control switch to the Toggle/Reset position to reset the actuator.
3. Then set the Control switch to the Latch position.

V. Toggle Mode: In the Toggle Mode the Model 1640 allows the sampler to take samples only while the liquid is touching the probe assembly. The sampler will continue taking samples only as long as the liquid touches the stainless steel ring. If the liquid level recedes, the sampler will be inhibited until the liquid level again rises to the probe assembly. If the liquid level recedes while the sampler is taking a sample, it will finish taking the sample before shutting off.

A. Programming the Toggle Mode (assuming that steps II, A, 1-6 have been completed)
1. Place the sampler pump switch in the AUTO position.
2. Set the Model 1640 control switch to the Toggle/Reset position.

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TREATMENT PLANT OPERATORS

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pH BUFFER VALUES FOR VARYING TEMPERATURES

4.01 pH Buffer

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

7.00 pH Buffer

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

9.18 pH Buffer

Temperature Range(°C)	pH 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

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WATER ANALYSIS

TOTAL ALKALINITY

I. Determine pH of sample with pH meter. Only those samples with a pH above 8.3 will have phenalphthalein alkalinity.

II. Reagents

A. Phenolphthalein Indicator

1. Phenolphthalein	5 g
2. Ethyl 95% or Isopropyl Alcohol	500 ml
3. Distilled Water	500 ml

Dissolve phenolphthalein in alcohol and Q.S. to 1000 ml with distilled water.

B. Bromcresol Green-Methyl Red Indicator

1. Methyl Red Sodium Salt	1.0 g
2. Bromcresol Green Sodium Salt	0.2 g
3. Distilled Water	Q.S. 1000 ml

C. 0.02N H_2SO_4 Titrant Solution (See Section VI for Standardization)

1. 1.0N H_2SO_4	40 ml
2. Distilled Water	Q.S. 2000 ml

D. 0.05N Sodium Carbonate Solution (for standardizing acid)

1. Na_2CO_3 (Dried at 250°C for 4 hours)	2.5 g
2. Distilled Water	Q.S. 1000 ml

3. Stable 1 week

III. Procedure

A. Sample volume	50 ml
B. Phenolphthalein Indicator	3 drops

1. Titrate sample with 0.02N H_2SO_4 from the pink color to colorless. Record mls of titrant x 20 as P. Alkalinity (example: 1.5 ml titrant used x 20 = 30 P. Alkalinity)

C. Bromcresol Green - Methyl Red Indicator 3 drops

1. Continue titrating with 0.02N H_2SO_4 from the green color to reddish pink. Record mls of titrant x 20 as Total Alkalinity (example: 6.5 ml titrant used x 20 = 130 total alkalinity)

IV. Carbonates as $CaCO_3$

A. Multiply p. alkalinity x 2 (example: 30 p. alkalinity x 2 = 60 carbonates as $CaCO_3$

V. Bicarbonates as $CaCO_3$

A. Subtract carbonates from Total Alkalinity (example: 130 total alkalinity - 60 carbonates = 70 bicarbonates as $CaCO_3$

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VI. Standardization of 0.02N H_2SO_4

A. Only titrant actually used in Lab must be standardized.
B. Using a volumetric pipet (Class A) transfer 15.00 ml of 0.05N Na_2CO_3 to a small beaker.
C. Add 60 ml of distilled water to beaker.
D. Titrant, using 0.02N H_2SO_4 down to a pH of 5.
E. Rinse electrodes into beaker.
F. Place a watch glass cover over beaker and boil 3 to 5 minutes.
G. Cool to room temperature, rinse cover glass into beaker.
H. Add Bromcresol Green-Methyl Red Indicator and titrate to end point (total~35 ml)
I. Calculations: If mls of acid used ≠ 35 normality of acid

$$\text{True N} = \frac{A \times 15.00}{53.00 \times B}$$

A = g Na_2CO_3 (~2.5g) B = ml acid (35 ml)

VII. Calculations (If normality of acid ≠ 0.02)

$$\text{Alkalinity} = \frac{A \times N \times 50{,}000}{50}$$

VIII. Alkalinity Relationship Table

Results	Hydroxide Alkalinity	Carbonate Alkalinity	Bicarbonate Alkalinity
P = 0	0	0	T
P<½T	0	2P	T-2P
P=½T	0	2P	0
P>½T	2P-T	2(T-P)	0
P=T	T	0	0

P = Phenolphthalein Alkalinity

T = Total Alkalinity

IX. References
A. Standard Methods, 15th Ed., Method 403
B. HACH DR/3000 Spectrophotometer Manual

X. Revised by: Gaines B. Huneycutt
Date: 9 September 1985

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CHLORIDES

I. Reagents:

A. Potassium Chromate Indicator (K_2CrO_2)

1. Potassium Chromate 50.0g
   a. Dissolve potassium chromate in about 100ml of distilled water. Add $AgNO_3$ 0.0282N (about 10ml) until a red parcipitate is formed Allow to stand for 12 hours. Filter and dilute filtrate to 1000ml.

B. Silver Nitrate ($AgNO_3$) 0.0282N

1. Silver Nitrate 4.790g (9.580g)
   a. Dissolve silver nitrate in 500ml (1000ml) distilled water. Q.S. to 1000ml (2000ml) with distilled water.
   b. Store in brown bottle to protect from light.

C. Sodium Chloride (NaCl) 0.0282N (for standardization)

1. Sodium Chloride 16.480g
   a. Dry NaCl for 1 hour at 140°C before weighing
2. Distilled Water Q.S. 1000ml

II. Standardization of 0.0282N Silver Nitrate

A. Only titrant actually used in Lab must be standardized
B. Using a volumetric pipet (Class A) transfer 50ml of 0.0282N Sodium Chloride to an erlemeyer flask
C. Fill another flask with 50ml of distilled water (Blank)
D. Add chromate indicator (2-3 drops) to both flasks and titrant from a yellow to a red with the 0.0282 $AgNO_3$
E. Calculations

A= mls of titrant in NaCl Flask B = mls of titrant in blank

1. If A-B = 2.5 ml no calculation required
2. If A-B ≠ 2.5 ml calculate true normality (N):

$$N = \frac{0.0705}{(A-B)}$$

III. Procedure:

A. 50ml sample.
B. Add 2-3 drops chromate indicator.
C. Titrate with 0.0282N $AgNO_3$ from chromate yellow to red.

IV. Calculations:

A. If N = 0.0282

$$mgCl/L = (mls\ AgNO_3 - B) \times 20$$

$$mgNaCl/L = (mgCl/L) \times 1.65$$

B. If N ≠ 0.0282

$$mgCl/L = (mls\ AgNO_3 - B) \times N \times 709$$

$$mgNaCl/L = (mgCl/L) \times 1.65$$

V. References:

A. Standard Methods, 15th Ed, Method 407A

VI. Revised by: Elizabeth A. Betz
Date: 12 September 1985

DETERMINATION of CaO in LIME

I. SAMPLE PREPARATION--
Grind about 50 grams of a well-mixed sample of the subject lime in a mortar and pestle until all of it is fine enough to pass through a No. 100 sieve.

II. REAGENTS--
A) Boiled and cooled (CO_2-free) distilled water. Used any time this procedure calls for "water". Keep covered to avoid contamination by CO_2 in air.

B) 0.1782 N standardized HCl.
PREPARATION:
Dry about five grams of Sodium Carbonate ($NaCO_3$) at 103°C for one hour.
Add 14.5 ml of concentrated ACS HCl to about 500 ml of water and QS to 1000 ml.

STANDARDIZATION:
Weigh 0.8500 gram of $NaCO_3$ and dissolve in 100 ml of water.
Titrate this with the acid, using ½ ml of Methyl Orange indicator.
Titrate this to a salmon-pink endpoint. If acid normality is exactly 0.1782, it will take 90.0 ml of acid.

CORRECTION ("K") FACTOR:
It it takes more or less than 90.0 ml of acid to reach the end point, determine the K-factor to use in calculating the final answer thus:

$$K = \frac{\text{Milliliters of acid used}}{90.0 \text{ ml}}$$

C) Phenolphthalein Solution (4%).
Dissolve 4 grams of phenolphthalein in 100 ml of 95% ethanol.

D) Cane sugar (Domino, Dixie Crystals, etc.)

III. PROCEDURE:
1. Weigh 0.5 gram of pulverized lime and add to a 250 ml flask containing 10 ml of water. Stopper immediately (loosely) and swirl to disperse particles.
2. Bring 50 ml of water to a boil, and add to the flask. Place flask on heat and boil for one minute. (This "slakes" the lime.)
3. Stopper loosely, and place in cold water bath to cool to room temp.
4. Add 50 ml of (room temp) water, then immediately add 16 grams of sugar.
5. Stopper, swirl, and let stand for 15 minutes, swirling every 5 minutes.
6. Add 5 drops of phenolphthalein, wash sides of flask with water, and titrate to colorless with 0.1782 N HCl.
(Add the first 50 ml of acid by volumetric pipette, without shaking the flask. Continue titration by 50 ml burette. Avoid excessive shaking until almost all of the acid has been added.)
7. Run in duplicate.

IV. CALCULATION:
Multiply the total milliliters of acid used by the acid "K" factor to give the percent available CaO in the lime sample.

V. REFERENCE:

A. American Waterwork Association (AWWA) Standard for Quicklime and Hydrated Lime, 30 January 1977

VI. Written by: A. T. Luke
Date: April 1981

DETERMINATION of CaO in LIME

I. SAMPLE PREPARATION--

Grind about 50 grams of a well-mixed sample of the subject lime in a mortar and pestle until all of it is fine enough to pass through a No. 100 sieve.

II. REAGENTS--

A) Boiled and cooled (CO_2-free) distilled water. Used any time this procedure calls for "water". Keep covered to avoid contamination by CO_2 in air.

B) 0.1782 N standardized HCl.

PREPARATION:

Dry about five grams of Sodium Carbonate ($NaCO_3$) at 103°C for one hour.
Add 14.5 ml of concentrated ACS HCl to about 500 ml of water and QS to 1000 ml.

STANDARDIZATION:

Weigh 0.8500 gram of $NaCO_3$ and dissolve in 100 ml of water.
Titrate this with the acid, using ½ ml of Methyl Orange indicator.
Titrate this to a salmon-pink endpoint. If acid normality is exactly 0.1782, it will take 90.0 ml of acid.

CORRECTION ("K") FACTOR:

It it takes more or less than 90.0 ml of acid to reach the end point, determine the K-factor to use in calculating the final answer thus:

$$K = \frac{\text{Milliliters of acid used}}{90.0 \text{ ml}}$$

C) Phenolphthalein Solution (4%).
Dissolve 4 grams of phenolphthalein in 100 ml of 95% ethanol.

D) Cane sugar (Domino, Dixie Crystals, etc.)

III. PROCEDURE:

1. Weigh 0.5 gram of pulverized lime and add to a 250 ml flask containing 10 ml of water. Stopper immediately (loosely) and swirl to disperse particles.
2. Bring 50 ml of water to a boil, and add to the flask. Place flask on heat and boil for one minute. (This "slakes" the lime.)
3. Stopper loosely, and place in cold water bath to cool to room temp.
4. Add 50 ml of (room temp) water, then immediately add 16 grams of sugar.
5. Stopper, swirl, and let stand for 15 minutes, swirling every 5 minutes.
6. Add 5 drops of phenolphthalein, wash sides of flask with water, and titrate to colorless with 0.1782 N HCl.
   (Add the first 50 ml of acid by volumetric pipette, without shaking the flask. Continue titration by 50 ml burette. Avoid excessive shaking until almost all of the acid has been added.)
7. Run in duplicate.

IV. CALCULATION:

Multiply the total mililiters of acid used by the acid "K" factor to give the percent available CaO in the lime sample.

COLORIMETRIC PROCEDURES

SPECTROPHOTOMETER GENERAL INSTRUCTIONS

A. Bausch & Lomb Spectronic 20
   1. Turn On: Turn power switch/zero control (see Figure 1) to the right until the pilot lamp comes on.
   2. Set Wavelength: Turn wavelength control (see Figure 1) to desired wavelength. Wavelength scale is located left on control.
   3. Cuvette Use
      a. Use B&L ½" test tubes with white index mark.
      b. Place in sample holder such that the white index mark aligns with the mark on the edge of the sample holder (under door).
   4. Set 0 % T
      a. Insert cuvette with required solution.
      b. Use power switch/zero control to adjust.
      c. Read top scale.
   5. Set 100% T
      a. Insert cuvette with required solution.
      b. Use 100% T control (see Figure 1) to adjust.
      c. Read top scale.

B. HACH DR/3000 Spectrophotometer
   1. Turn On: Power on switch is located at top right back of instrument.
   2. Set Wavelength: Turn wavelength control (see Figure 2) to desired wavelength. Wavelength digital readout is located right above control.
   3. Sample cell use.
      a. Use paired sample cells.
      b. Place in cell holder located under door so that the 25 ml fill mark on cell faces the front of the instrument.
      c. Even paired cells are not exactly the same so when possible use the same cell throughout the whole test.

C. General
   1. Sample holder doors must be closed to obtain readings.
   2. Sample cells or cuvettes are not to be used if:
      1. Discolored.
      2. Scratched, etched.
   3. All sample cells or cuvettes will be wiped with kimwipes just prior to being inserted into holder, to remove finger prints which could affect results.

FLUORIDE

ELECTRODE METHOD PROCEDURE

I. Apparatus, Reagents and Standards
- A. Fisher Accumet Ion-Selective Meter Model 750 w/Switch
- B. Fluoride Probe
- C. Stock Fluoride Solution (100 ppm)
  - 1. Anhydrous Sodium Fluoride (NaF) — 221 mg
  - 2. Distilled Water — Q.S. 1000 ml
- D. 0.1 ppm Standard w/TISAB
  - 1. Stock Fluoride (100 ppm) — 0.5 ml
  - 2. Distilled Water — Q.S. 500 ml
  - 3. TISAB — 500 ml
- E. 2.0 ppm Standard w/TISAB
  - 1. Stock Fluroide (100 ppm) — 20 ml
  - 2. Distilled Water — Q.S. 1000 ml
  - 3. TISAB — 1000 ml
- F. Total Ionic Strenght Adjuster (TISAB)
  - 1. In a 2 Liter Beaker Mix
    - a. Distilled Water — 1000 ml
    - b. Glacial Acetic Acid — 114 ml
    - c. Sodium Chloride — 116 g
    - d. CDTA — 8 g
  - 2. Stir above to dissolve, solution will be somewhat milky.
  - 3. Place a pH electrode into solution and add 5N NaOH to adjust to pH 5.0-5.5 (approx. 300 ml).
  - 4. Cool solution, dilute to 2 liters in a volumetric flask.

II. CAUTION: METER MUST BE IN STANDBY BEFORE PULLING ELECTRODE OUT OF SOLUTION.

III. Meter and Switch Preparation
- A. Meter should be in Standby at switch and meter.
- B. With meter in Standby, place the temperature probe with the F- probe in F- buffer.
- C. Turn knob on Switch to "2" (F-).
- D. Press STBY/MEAS on meter until in Standby.
- E. DO NOT bother with the Switch anymore.
- F. Proceed to Standardization or Samples.

IV. Standardization (Weekly or as required)
- A. Press Mode until in Concentration.
- B. Press STBY/MEAS until out of Standby.
- C. If not in Standardize, Press STDZ/PROG.
- D. Return to Standby (Press STBY/MEAS).
- E. Place Electrodes in 0.1 ppm, press STBY/MEAS.
- F. Enter 0.1000, and press ENTER.
- G. When millivolt readings stabilizes, 3 min (may take 5 min), press ENTER again.
- H. Return to Standby, press STBY/MEAS.
- I. Place electrode in 2.00 ppm, press STBY/MEAS. Repeat steps 6-8, entering 2.0000.

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V. Sample Handling
   A. Collect at least 100 ml of sample.
   B. Pour 50 ml or 15 ml of sample into small beaker.
   C. Add an equal amount of TISAB to beaker.

VI. Running Samples
   A. Place in Standby, press STBY/MEAS, rinse electrodes and place in sample.
   B. Press STBY/MEAS, wait 3 min. Read result when stabilized.

VII. Shut Down
   A. Press STBY/MEAS until meter is in Standby.
   B. Place all probes in appropriate buffers.
   C. Turn knob on Switch to "Standby".

VIII. References

   A. Standard Methods, 15th Ed., Method 413B.
   B. EPA Methods for Chemical Analysis of Water and Wastes, March 1983, Method 340.1.
   C. Instruction Manual for Fisher Accumet Model 750 Selective Ion Analyzer.

IX. Revised by: Elizabeth A. Betz
   Date: 2 October 1985

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STABILITY TEST

I. Equipment and Reagents:
   1. pH meter.
   2. Precipitated calcium carbonate.
   3. Sample cup (150 ml beaker).
   4. Filter paper

II. Test Procedures:
   1. Measure pH of sample (same as a regular pH test). This is pH_a (pH actual).
   2. Add precipitated calcium carbonate (in excess) to sample, stir a few minutes, allow to settle then filter supernatant. (A few grams will be needed to make an excess - you know you have an excess if all the powder will not dissolve).
   3. Measure pH of the filtered sample. This is pH_s (pH saturated).
   4. Calculations.
      a. Stability = $pH_a - pH_s$

III. Interpretation:
   1. Positive values indicate scale forming.
   2. Negative values indicate corrosive.
   3. Zero values indicate neutral or stable water.

IV. Plant operating range:
   1. Best range - from zero to + 1.0

V. Revised by: Elizabeth A. Betz
   Date: 28 August 1985

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water

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STABILITY TEST - WATER TREATMENT

I. Sampling:

1. Collect a representative sample delivered water.

2. Run as soon as possible.

II. Equipment supplied by Utilities Branch, BMAINDiv:

1. pH meter

2. pH Probe (on meter)

III. Equipment and reagents supplied by Quality Control Lab, Environmental Branch, NREADiv:

1. Precipitated calcium carbonate

2. Sample cup (150 ml beaker)

3. Filter paper

IV. Test Procedures:

1. Measure pH of sample (same as regular pH test). This is pH_a (pH actual).

2. Add precipitated calcium carbonate (in excess) to sample, stir a few minutes, allow to settle then filter supernatant. (A few grams will be needed to make an excess - you know you have an excess if all the powder will not dissolve).

3. Measure pH of the filtered sample. This is pH_s (pH saturated).

4. Calculations.

   a. Stability = $pH_a - pH_s$

V. Interpretation:

1. Positive values indicate scale forming.

2. Negative values indicate corrosive.

3. Zero values indicate neutral or stable water.

VI. Plant operating range:

1. Best range - from zero to + 1.0

2. If you have any problems, see your Leader or Foreman

pH Test-Water Treatment

Orion Model 301

I. Sampling
1. Collect a representative sample of about 200 mls.
2. Run the pH as soon as possible.

II. Equipment-Supplied by Utilities Branch, BMaintDiv
1. pH Meter
2. pH Combination Electrode
3. Thermometer in degrees centigrade(Celsius)

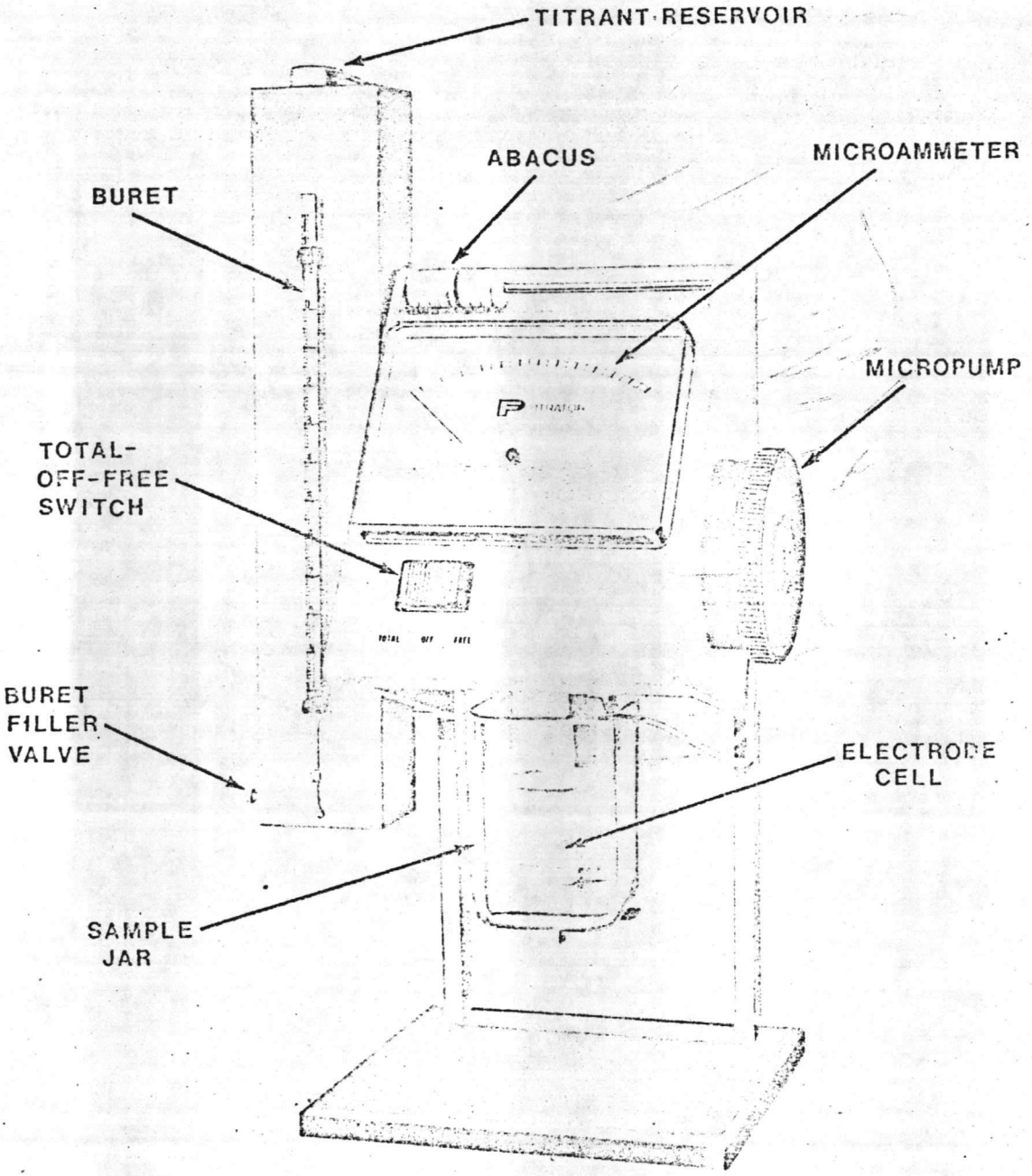
III. Reagents and Supplies-Supplied by Quality Control Lab, Envir. Br., NREADiv
1. pH Buffer Solutions
   a. pH 9.18
   b. pH 7.00
2. Distilled Water-for washing electrode and sample beaker
3. Wash Bottle-for Distilled Water
4. Sample Beaker

IV. Instrument Set Up
1. If applicable, check level of electrolite in probe and add as necessary to keep full with pH Combination Electrode Filling Solution.
2. Always leave electrode in distilled water or pH buffer between tests.

V. Meter Calibration
1. Measure temperature of the 9.18 pH buffer solution
2. Set temperature knob on pH meter to the pH buffer solution temperature.
3. Rinse electrode with distilled water and place electrode in a fresh beaker of 9.18 pH buffer solution.
4. Swirl the solution several times and allow meter needle to stabilize.
5. Adjust the needle on the reference scale by using the calibration knob on pH meter to the correct pH reading as indicated on the chart below for 9.18

VI. Verify Calibration
1. Measure temperature of the 7.00 pH buffer solution.
2. Set temperature knob on pH meter to the pH buffer solution temperature.
3. Rinse electrode with distilled water and place electrode in a fresh beaker of 7.00 pH buffer solution.
4. Swirl the solution several times and allow meter needle to stabilize.
5. Read pH. pH reading should be within 0.2 of the true pH value as indicated on the chart below for 7.00. If it is not, one or both buffers, probe or meter maybe bad.

VII. pH Determination of Sample
1. Calibrate meter and verify that it is working properly.
2. Measure temperature of sample.
3. Adjust temperature control knob to that of the sample.
4. Wash the electrode with distilled water and immerse the electrode into the sample.
5. Swirl the sample several times and allow the needle to stabilize before reading the pH value on the meter.
6. Record meter reading on log sheet.
7. Remove electrode form sample, wash with distilled water and place electrode in distilled water or pH buffer solution.


TITRANT RESERVOIR
ABACUS
MICROAMMETER
BURET
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Free Residual Chlorine Test-Water Treatment

Amperometric Titration Method

I. Sampling
1. Collect a representative sample of about 200 mls.
2. Perform the test as soon as possible after the sample is collected.

II. Equipment and Reagents-Supplied by Quality Control Lab, Envir. Br., NREADiv
1. Fischer & Porter Model 17T1010 Amperometric Titrator
2. 200 ml sample cup (comes with titrator)
3. Dropper Bottle of pH 7 Buffer
4. Phenylarsine oxide (PAO)titrant
5. Automatic Glass Titrator (attached to Amperometic Titrator)

III. Procedure
1. Fill glass titrator with PAO and zero.
2. Clean out sample cup and place 200 mls of sample in it.
3. Add 1 eyedropper of pH 7 Buffer.
4. Place sample cup on titrator.
5. Place the switch on the FREE position.
6. Make sure the stopcock on the glass titrator is open.
7. Slowly rotate the micro pump clockwise to titrate.
8. Continue to add titrant drop by drop until the addition of one more drop does not cause the needle to move to the left.
9. If the level in the glass titrator reaches 10 before the needle stops moving, rezero the titrator and move one abacus on top to the right.
10. When the needle stops moving read the mls off the glass titrator, add 10 mls for every abacus on the right. The total mls is equal(=) to ppm of total residual chlorine.
11. Record the total residual chlorine in the log.
12. Place the switch in OFF. Leave the sample in the sample jar and leave it on the titrator. Close stopcock on glass titrator.

IV. Comments
1. This is only a back up procedure when DPD tablets are not available.
2. The Quality Control Lab has 3 Amperometric Titrators for use. They are Lab property and will be returned when DPD becomes available.
3. If there is any problem with this procedure or equipment, call the Lab (5977). Do Not Mess with it.

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FREE RESIDUAL CHLORINE TEST - WATER TREATMENT

DPD SOLUTIONS COMPARATOR 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

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

A. Glass Stirring Rods

B. Distilled Water

C. Eyedroppers (2)

D. DPD Buffer

E. DPD Solution

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 ½ ml (fill eyedropper to line) of DPD Buffer to one tube.

D. To same tube add ½ ml (fill eyedropper to line) of DPD Solution.

E. Place this tube with tablet in the right side of the comparator.

F. Place the other tube (just sample, no DPD) in the left side.

G. Read the ppm chlorine by comparing the colors of the chlorine disc to the sample within 3 minutes.

H. Record the ppm residual chlorine on the Bacti sheet or Bacti bottle.

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to calibrate the meter

For meters with an automatic temperature compensation: Proceed to Step Three

2. Set temperature knob on pH meter to the pH buffer solutions temperature.
3. Rinse electrode with distilled water and place in a fresh beaker of the second buffer solution.
4. Swirl the solution several times and allow meter to stabilize.
5. Read pH. pH reading should be within 0.2 of the true pH value of the second buffer. If it is not, one or both buffers, probe(s) or meter may be bad.

VII. pH Determination of Sample

1. Calibrate meter and verify that it is working properly.
2. For meters without internal temperature compensation: Measure temperature of the sample.

For meters with an automatic temperature compensation: Proceed to Step Four

3. Set temperature knob on pH meter to the sample temperature.
4. Rinse electrode with distilled water and place in sample.
5. Swirl the sample several times and allow meter to stabilize.
6. Read pH. Record the meter reading on log sheet.
7. Remove electrode from sample, wash with distilled water and place electrode in distilled water or pH buffer solution.

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COLLECTION OF BACTERIA SAMPLES FROM SWIMMING POOL - TARAWA TERRACE

1. Bacteria samples will be collected from the T.T. pool from two locations. (Extra samples)

   a. Take one sample from the deep end. Place both feet on the deep sign located next to the life guard station directly out of the filter room.

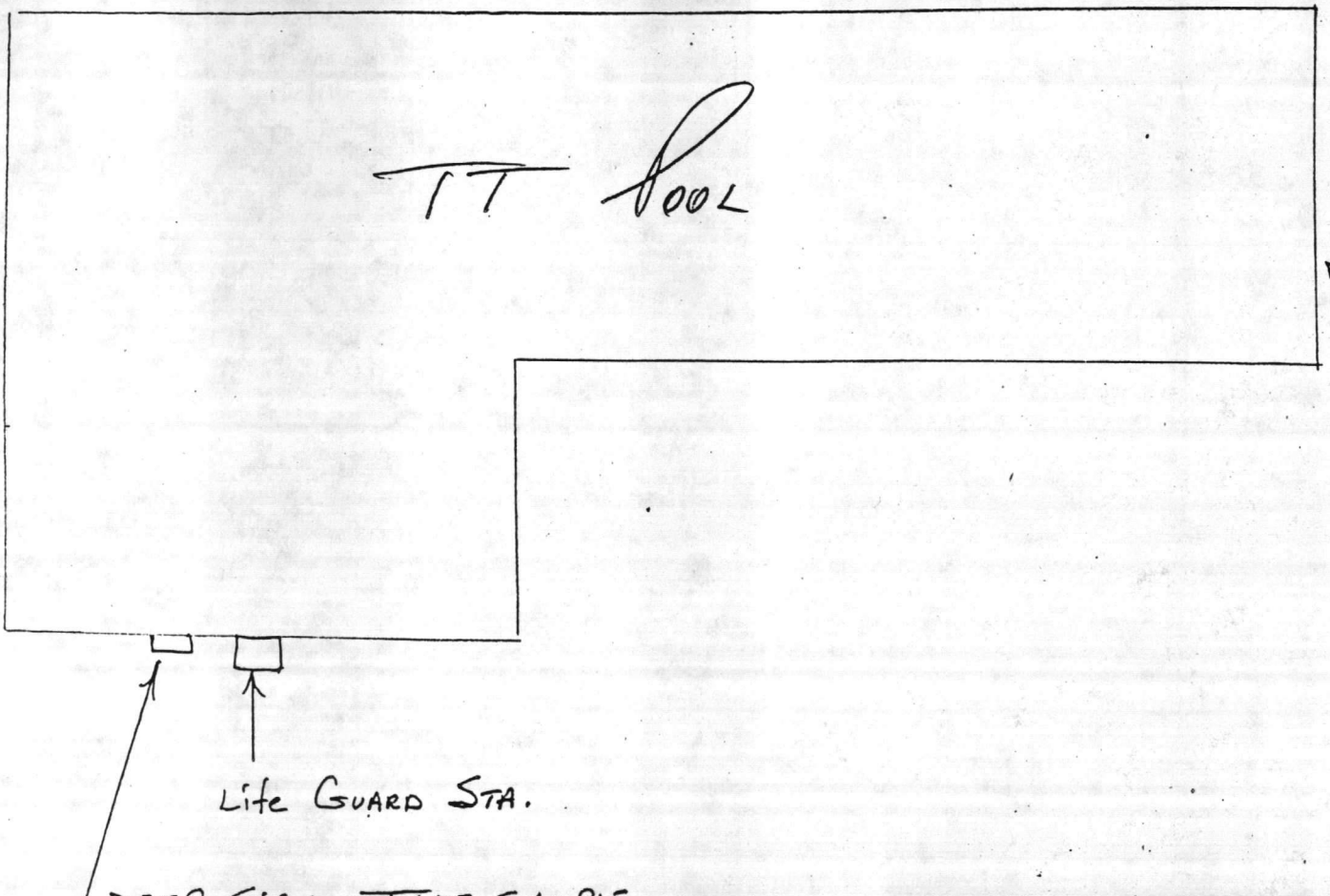
   b. Collect the other sample from the shallow end of the pool between the #2 and #3 diving platforms. Use correct procedures as outlined below.

PROCEDURES FOR COLLECTION OF SAMPLES

1. Take and record chlorine, p.H., and time sample taken, Record same on bottle or MCBCL 11330/4 if provided.

2. Sampling from pool - When collecting samples from standing water, remove the stopper as above, and plunge the bottle, mouth down and held at about a 45° angle at least 3 inches below the surface. Tilt bottle and allow air to escape and fill, moving it in a direction away from the hand holding it, so that water that has touched the hand does not enter the bottle. Discard a quarter of the water and replace the stopper.

3. Return to laboratory for analysis.



WASTEWATER TREATMENT OPERATOR'S NOTEBOOK

Table of Contents

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29 Sep 83

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

NREAD/DDS/th
11345

~~30 Sep 1983~~
30 Oct 83

From: Assistant Chief of Staff, Facilities
To: Base Maintenance Officer

Subj: Quality Control of Wastewater in-plant Sampling and Analysis Required by Federal and State Regulations

Ref: (a) National Pollutant Discharge Elimination System (NPDES) Permit of 26 Feb 1980
(b) Maintenance Order 11330.2

Encl: (1) Directory of Wastewater Treatment Plant Operating Personnel
(2) pH Test Procedure for Orion Model 301 Meter
(3) Total Residual Chlorine DPD Test Procedure
(4) Coliform Sampling Procedure
(5) Composite Sampling Procedure for Influent/Effluent

1. Wastewater Treatment Plant personnel are currently required to perform the following sampling and analysis in accordance with reference (a):

a. pH of Effluent (using meter)

b. Total Residual Chlorine of Effluent

c. Total Daily Flow

d. Effluent Coliform (Grab Sampling Only)

e. Influent and Effluent (Composite Sampling Only)

Data generated is reported quarterly to the Environmental Protection Agency and the NC Department of Natural Resources and Community Development.

2. NREAD is currently developing procedures for providing adequate quality control related to the subject sampling and analysis. Reference (b) pertains. Procedures will address accuracy and availability of written procedures for use by operators; availability and condition of equipment and supplies; and documenting ability of personnel to perform procedures. Procedures will provide on-site inspections of sewage treatment plants by NREAD personnel and use of automatic samplers on a spot check basis.

3. Because of the number and working hours of wastewater treatment operators, the cooperation of Utilities' managers is essential in ensuring operators are properly trained and proficient in the subject areas. It is requested that enclosure (1) be completed and returned by 15 October 1983. NREAD personnel will assist in providing training requirements identified by the Utilities Branch.

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NREAD/DDS/th
11345

Subj: Quality Control of Wastewater in-plant Sampling and Analysis Required by Federal and State Regulations

Enclosures (2), (3), (4) and (5) provide procedures addressed by enclosure (1) which plant personnel must be qualified to perform. It is requested that Utilities personnel review enclosures (2) - (5) and make any comments by 15 October 1983.

4. Enclosure (5) will require the operators to be provided a sample size chart for use in proportioning samples. It is requested that the hourly flow data for each plant be made available to NREAD personnel for the purpose of preparing this chart. NREAD personnel will review the data on-site with Utilities personnel, therefore, it is not necessary to furnish actual copies. It is requested that procedures in enclosures (2)- (5) be fully implemented not later than 1 November 1983. This will require revision of reference (b).

5. Input from Utilities Branch personnel in the subject area is encouraged, particularly with the development of a checklist to be utilized during in-plant inspections by NREAD. The point of contact for this is Mr. Danny Sharpe extensions 2083/1690.

M. G. LILLEY

Copy to:
Supvy Chemist (NREAD)

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Directory of Wastewater Treatment Plant Operating Personnel

Operator Name:	Sampling and Analysis		Sampling Only	
	pH (using meter)	Total Chlorine (by DPD)	Coliform	Composite Influent/Effluent

Note 1. Enter "NA" for any test employee not required to perform.

Note 2. Enter "X" for each procedure certified to perform.

Note 3. Leave blank where additional training required.

I hereby certify that the above employees are qualified to perform the analysis and sampling required by the NPDES permit as indicated above.

NAME _____ DATE _____

ENCLOSURE (1)

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MAIN/FEC/rn
11345

OCT 1 7 1983

From: Base Maintenance Officer
To: Assistant Chief of Staff, Facilities

Subj: Wastewater Sampling and Analysis

Ref: (a) AC/S, Fac ltr NREAD/DDS/th 11345 of 3 Oct 1983
(b) Maintenance Order 11330.2

Encl: (1) Directory of Wastewater Treatment Plant Operating Personnel

1. Reference (a) outlined various modifications to the existing wastewater sampling procedures. Base Maintenance agrees with the improvements to the sampling procedures and is already taking steps to implement them.

2. It should be noted that although the suggested changes will clarify and improve sampling procedures, previous procedures used for sampling complied with our NPDES Permit and were considered adequate by state and other regulatory personnel.

3. As requested by the reference, the enclosure provides a listing of the employees presently performing wastewater sampling. All but five of the employees listed have a Grade II certification (or higher) from the State of North Carolina to operate wastewater plants. Operator certification requires knowledge of sampling and laboratory techniques. All employees listed have been trained in sampling/analysis procedures and are considered qualified to perform sampling and analysis required by the NPDES Permit. However, to our knowledge, no specific certification exists for sampling/analysis procedures. It is recommended that NREAD personnel provide specific training for any new procedures approved and begin monthly visits to sewage treatment plants in accordance with reference (b), the existing order, to assist plant personnel with sampling procedures.

4. In addition, it is requested that any changes in procedure that are noted during plant visits by NREAD personnel be approved by the Utilities Director prior to implementation to insure required transmittal to all employees. Likewise, it is requested that any procedural errors, problems with employees or any other situations that adversely affect the quality of sampling be reported directly to the Utilities Director for appropriate action.

5. Further, it is recommended that a meeting be scheduled with NREAD and Base Maintenance personnel to discuss any additional procedures that will improve wastewater quality control. Point of contact for Base Maintenance

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MAIN/FEC/rn
11345

Subj: Wastewater Sampling and Analysis

is Mr. F. E. Cone, extension 5161.

J. T. MARSHALL

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TABLE 5

DIRECTORY OF APPROVED WASTEWATER PLANT OPERATORS FOR SAMPLE COLLECTION

ALDRIDGE, Barry T.
AMBROSE, John H.
ANTINORI, David L.
BROWN, Clennie L.
CARLYLE, Billy B.
COLLINS, Edward G.
~~CONNOR, Joe A.~~
CREWS, Stephen V.
~~DARDEN, Glenn~~ L.
DAVILA, Gabriel
DAVIS, Mack D. Jr.
DELGADO-NIEVES, Dolores
FARLAND, Melvin S.
FARROW, McArthur
FUTREAL, Rupert
FUTRELL, Norvin J.
GODWIN, Otis E.
HALL, Leitha W.
HILL, Stanley E.
HUDGINS, Alton O.
KELLUM, Kenneth D.
KENNEDY, Tommie H.
NORRIS, Rebecca E.
PACK, Donald L.
PATE, James C.
RHODES, Randal B.
ROLLINGER, David L.
SARDINAS, Frank
SAULTER, Albert F. Jr.
~~SCHMIDT, Carroll V.~~
SNODGRASS, Anthony P.
SNODGRASS, Pamela C.
STEVENSON, David M.
TAYLOR, Herman B.
TAYLOR, Johnnie P.
THOMPSON, James L.
TREDWELL, David H.
WILLIAMS, Victor W.
WOOLDRIDGE, Earl C.
YOPP, Everett D.

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pH Test-Wastewater Treatment

Orion Model 301

I. Sampling

A. Collect a representative sample of about 200 mls

B. Run the pH as soon as possible

II. Equipment-Supplied by Utilities Branch, Base Maintenance Division

A. pH Meter

B. pH Combination Electrode

C. Thermometer in Degrees Centigrade (Celsius)

III. Reagents and Supplies-Supplied by Quality Control Laboratory, Environmental Branch, Natural Resources and Environmental Affairs Division

A. pH Buffer Solutions

1. pH 7.00

2. pH 4.01

3. pH 9.18

B. Distilled Water for Washing Electrode and Sample Beaker

C. Wash Bottle for Distilled Water

D. Sample Beaker

IV. Meter Calibration

A. Measure temperature of 7.00 pH buffer solution

B. Set temperature knob on pH meter to the pH buffer solution temperature

C. Rinse electrode with distilled water and place electrode in a fresh beaker of 7.00 pH buffer solution

D. Swirl the solution several times and allow meter needle to stabilize

E. Adjust the needle on the reference scale by using the calibration knob on pH meter to the correct pH reading as indicated on Attachment A for 7.00

V. pH Determination of Sample

A. Calibrate meter and verify that it is working properly

B. Measure temperature of sample

C. Adjust temperature control knob on meter to that of the sample

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ENCLOSURE (2)

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D. Wash the electrode with distilled water and immerse the electrode into the sample

E. Swirl the sample several times and allow the needle to stabilize before reading the pH value on the meter

F. Record meter reading on Attachment B. OF EFFLUENT SAMPLES.

G. Remove electrode from sample wash with distilled waster and place electrode in distilled water or pH buffer solution.

VI. Verify Calibration

A. Measure temperature of the 4.01 or 9.18 pH buffer solution which ever is closer to the sample.

B. Set temperature knob on pH meter to the pH buffer solution temperature

C. Rinse electrode with distilled water and place electrode in a fresh beaker of pH buffer solution

D. Swirl the solution several times and allow meter needle to stabilize

E. Read pH; pH reading should be within 0.2 of true pH value as indicated on Attachment A for the buffer. If it is not, one or both buffers, probe or meter may be bad.

VII. Reporting. Submit Attachment B to Quality Control Laboratory by third day of following month.

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pH Buffer Values for Varying Temperatures

4.01 pH Buffer

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

7.00 pH Buffer

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

9.18 pH Buffer

Temperature Range (°C)	pH 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

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pH Readings Using Meter

NOV 1 1983

Plant: ______________________

Date	Time	Name	Value	Date	Time	Name	Value

ATTACHMENT B

ENCLOSURE (2)

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Total Residual Chlorine Test-Wastewater Treatment

DPD Tablet Comparator 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.

L. Read the ppm chlorine be 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. Reprot 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 neutrlize chlorine. Do not rinse out. Additional bottles are available at WQCL if accidents occur.

11

ENCLOSURE (4)

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 form 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 BOD_5 and suspended solids analysis. (See note 1)

A. PLANT: Hadnot Point

B. SAMPLING FREQUENCY: Sunday, Tuesday, Wednesday, Thursday and Friday

C. SAMPLING PERIOD: 0001-2400 hours

D. SAMPLING INTERVAL: Once every hour

E. FLOWMETER: Use data routinely gathered at S-21 at foot of hill to determine "TOTAL HOURLY FLOW" (See note 2)

F. SAMPLE SIZE CHART:

Total Hourly Flow	Volume of Wastewater to be collected
000 - 100	50 ml
101 - 200	100 ml
201 - 300	150 ml
301 - 400	200 ml
401 - 500	250 ml
501 -	300 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.

Note 2

Add second and fourth column of the yellow pad together. The sum of these two columns is total hourly flow to be used in F above.

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SAMPLE SIZE CHART

Composite sampling information for #1,2 and 3 samples gathered for BOD_5 and suspended solids analysis. (See note 1)

A. PLANT: Tarawa Terrace

B. ROUTINE SAMPLING DAYS: Tuesday, Wednesday, Thursday and Friday

C. SAMPLING PERIOD: 0001-2400 hours

D. SAMPLING INTERVAL: Once every hour

E. FLOWMETER: Use meter located in office to determine Gauge on glass reading required below.

F. SAMPLE SIZE CHART:

Gauge on Glass Reading	Volume of Wastewater to be collected
0.0 - 0.5	50 ml
0.5 - 1.0	100 ml
1.0 - 1.5	150 ml
1.5 - 2.0	200 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.

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SAMPLE SIZE CHART

Composite sampling information for #1,2 and 3 samples gathered for BOD_5 and suspended solids analysis. (See note 1)

A. PLANT: Camp Geiger

B. ROUTINE SAMPLING DAYS: Tuesday, Wednesday, Thursday and Friday

C. SAMPLING PERIOD: 0001-2400 hours

D. SAMPLING INTERVAL: Once every hour

E. FLOWMETER: Use meter located in office to determine chart paper reading required below.

F. SAMPLE SIZE CHART:

Charter Paper Reading	Volume of Wastewater to be collected.
0 - 300	50 ml
301 - 600	100 ml
601 - 900	150 ml
901 -	200 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.

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SAMPLE SIZE CHART

Composite sampling information for #1,2 and 3 samples gathered for BOD_5 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.

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SAMPLE SIZE CHART

Composite sampling information for #1, 2 and 3 samples gathered for BOD_5 and suspended solids analysis (See Note #1).

A. PLANT: Rifle Range

B. SAMPLING FREQUENCY: Tuesday and Thursday

C. SAMPLING PERIOD: 0800-1600 Hours

D. SAMPLING INTERVAL: Once every hour

E. FLOWMETER: Use meter located in office to determine chart paper reading required below.

F. SAMPLE SIZE CHART: Flow Chart Paper #FRP 211G039 From 0 - 450

Chart Paper Reading	Volume of Wastewater to be collected
0-100	125 ml
101-200	250 ml
201-300	375 ml
301-400	500 ml
401-	625 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.

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SAMPLE SIZE CHART

Composite sampling information for #1, 2 and 3 Samples gathered for BOD_5 and suspended solids analysis. (See Note #1)

A. PLANT: Courthouse Bay

B. ROUTINE SAMPLING DAYS: Tuesday and Thursday

C. SAMPLING PERIOD: 0800 - 1600 hours

D. SAMPLING INTERVAL: Once every hour

E. FLOWMETER: Use meter located in office to determine chart paper reading required below.

F. SAMPLE SIZE CHART:

Chart Paper Reading	Volume of Wastewater to be collected
0 - 100	100 ml
101 - 200	200 ml
201 - 300	300 ml
301 - 400	400 ml
401 -	500 ml

NOTE #1:

Please contact your supervisor/leader immediately for guidance if problem encountered relative to using flowmeter 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.

ENCLOSURE (1)

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SAMPLE SIZE CHART

Composite sampling information for #1,2 and 3 samples gathered for BOD_5 and suspended solids analysis. (See note 1)

A. PLANT: Onslow Beach

B. ROUTINE SAMPLING DAYS: Tuesday and Thursday

C. SAMPLING PERIOD: 0800 - 1600 hours

D. SAMPLING INTERVAL: Once every hour

E. FLOWMETER: Use meter located in office to determine "Gauge on Glass Reading" requried below.

F. SAMPLE SIZE CHART:

Gauge on Glass Reading	Volume of Wastewater to be collected
0 - 100	125 ml
101 - 200	250 ml
201 - 300	375 ml
301 - 400	500 ml

Note 1

Please contact 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.

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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 contatiners 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.

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DISSOLVED OXYGEN TEST-WASTEWATER TREATMENT

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. Manganeous 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 manganeous 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.

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9/08

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.

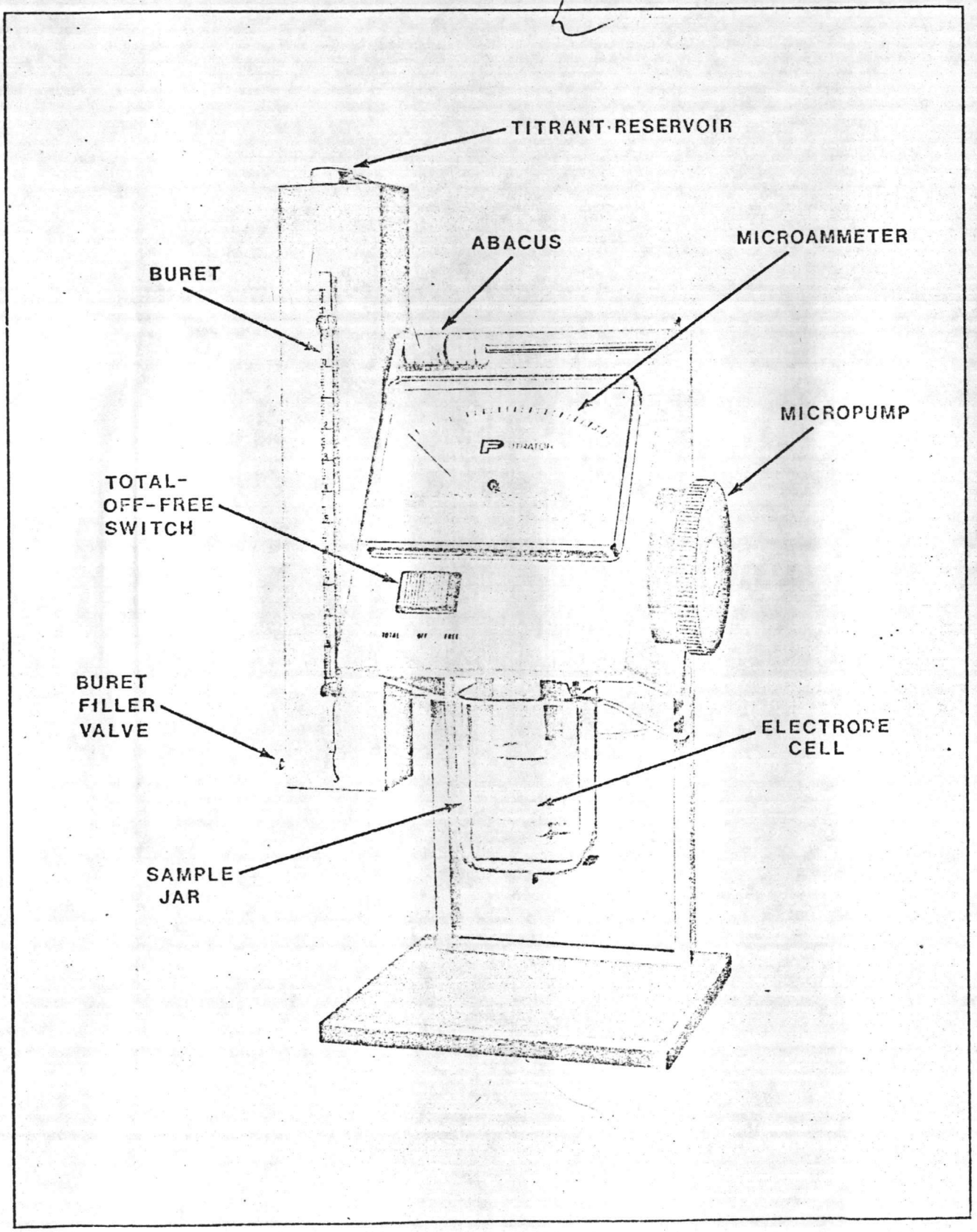
Total Residual Chlorine Test-Wastewater Treatment
Amperometric Titration Method

I. Sampling
1. Collect a representative sample of about 200 mls.
2. Perform the test as soon as possible after the sample is collected.

II. Equipment and Reagents-Supplied by Quality Control Lab, Envir. Br., NREADiv
1. Fischer & Porter Model 17T1010 Amperometric Titrator
2. 200 ml sample cup(comes with titrator)
3. Dropper Bottle of pH 4 Buffer
4. Dropper Bottle of 5% KI
5. Phenylarsine oxide (PAO) titrant
6. Automatic Glass Titrator(attached to Amperometic Titrator)

III. Procedure
1. Fill glass titrator with PAO and zero.
2. Clean out sample cup and place 200 mls of sample in it.
3. Add 1 eyedropper of pH 4 buffer.
4. Add 1 eyedropper of 5% KI.
5. Place sample cup on titrator.
6. Place the switch on the TOTAL position.
7. Make sure the stopcock on the glass titrator is open.
8. Slowly rotate the micro pump clockwise to titrate.
9. Continue to add titrant drop by drop until the addition of one more drop does not cause the needle to move to the left.
10. If the level in the glass titrator reaches 10 before the needle stops moving, rezero the titrator and move one abacus on top to the right.
11. When the needle stops moving read the mls off the glass titrator, add 10 mls for every abacus on the right. The total mls is equal(=) to ppm of total residual chlorine.
12. Record the total residual chlorine in the log.
13. Place the switch in OFF. Leave the sample in the sample jar and leave it on the titrator. Close stopcock on glass titrator.

IV. Comments
1. This is only a back up procedure when DPD tablets are not available.
2. The Quality Control Lab has 3 Amperometric Titrators for use. They are Lab property and will be returned when DPD becomes available.
3. If there is any problem with this procedure or equipment, call the Lab (5977). Do Not Mess with it.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Total Residual Chlorine Test-Wastewater Treatment

Ortho-tolidine Comparator Method

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

II. Equipment-Supplied by Utilities Branch, BMaintDiv
   1. Hellige Color Comparator
   2. Precision Tubes(2)

III. Equipment and Reagents-Supplied by Quality Control Lab, Envir. Br., NREADiv
   1. Hellige Ortho-tolidine Chlorine Color Disc.
   2. Ortho-tolidine Reagent

IV. Procedure
   1. Fill both sample tubes to the 10 ml mark.
   2. Add 0.5 ml of ortho-tolidine to one of the sample tubes, mix and place in the right side of the comparator.
   3. Place the other tube in the left side of the comparator.
   4. Wait five minutes for the color to develop.
   5. Read the ppm chlorine by comparing the colors of the chlorine disc to the sample.

V. Comments
   1. SAFETY NOTE: ORTHO-TOLIDINE IS POISONOUS, TOXIC AND CARCINOGENIC. DO NOT ALLOW IT TO TOUCH YOUR SKIN OR INHALE THE FUMES.
   2. This is only to be used when DPD tablets are not available. The Quality Control Lab will not release the discs or reagents without clearance from your leader or foreman.