5221

# State of North Carolina
## Department of Natural Resources and Community Development
Division of Environmental Management
512 North Salisbury Street • Raleigh, North Carolina 27611

James G. Martin, Governor
S. Thomas Rhodes, Secretary

February 19, 1987

R. Paul Wilms
Director

Ms. Elizabeth Betz, Supervisor Chemist
Natural Resources and Environmental Affairs Div.
Assistant Chief of Staff
Camp Lejeune, NC 28542

Dear Ms. Betz:

We have evaluated results from your analysis of wastewater laboratory certification performance samples received on February 18, 1987. The information from this evaluation is summarized below:

| Parameter | Value Reported | True Value | State Lab Value | Range | Performance |
|---|---|---|---|---|---|
| Total Phosphorus | 0.12 mg/1 | 0.118 mg/1 | | | Satisfactory |

(X) No additional follow-up is required as all results are acceptable.
( ) Another sample(s) is included for your analysis.
( ) You will be provided another sample at a future date.
(X) You will be contacted to establish a date for your on-site laboratory inspection.
( ) We will issue certification upon receipt of payment for the enclosed invoice.

Contact us at 919-733-3908 if you have questions concerning this matter.

Sincerely,

Billy O. Byrd

Laboratory Section

cc: Ted Cashion
Regional Supervisor, WiRO

6740/3
~~NREAD (L)~~
17 Feb 87

Mr. W. B. Edwards
NCRD/DEM Laboratory Branch
Post Office Box 27687
Raleigh, North Carolina 27611

Dear Sir:

In accordance with your 19 January 1987 letter, the Certified Laboratory Performance Analysis Report is submitted. Questions regarding this report should be forwarded to Ms. Elizabeth Betz, Supervisory Chemist, Natural Resources and Environmental Affairs Division, Assistant Chief of Staff, Facilities at (919) 451-5977.

Sincerely,

J. I. WOOTEN
Director

Encl:
(1) Certified Laboratory Performance Analysis Report

Copy to:
ECML, NREAD (2)

N 11987

Cert No _____

## Certified Laboratory Performance Analysis Report

This form is to be used to report the results of your analysis of the enclosed performance evaluation samples. One completed copy of this form is the only report needed. Do not return the individual report forms or mailing cartons. In the appropriate space below, enter the dilution made on the ampul contents (example: 5/1000 for 5 ml to 1000 ml), the value obtained from your analysis, and return this report to:

NRCD/DEM
Laboratory Section
P. O. Box 27687
Raleigh, NC 27611-7687

within 30 days of your receiving date. Reports received after this 30 day period will be considered unsatisfactory.

| Parameter | Dilution Made | Value Obtained | Parameter | Dilution Made | Value Obtained |
|---|---|---|---|---|---|
| BOD | _____ | _____ mg/l | Metals, Group II: | | |
| COD | _____ | _____ mg/l | Antimony | _____ | _____ µg/l |
| Chloride | _____ | _____ mg/l | Silver | _____ | _____ µg/l |
| Cyanide | _____ | _____ mg/l | Thallium | _____ | _____ µg/l |
| Fluoride | _____ | _____ mg/l | Arsenic | _____ | _____ µg/l |
| Grease & Oil | _____ | _____ mg/l | Barium | _____ | _____ µg/l |
| Hardness | _____ | _____ mg/l | Mercury | _____ | _____ µg/l |
| MBAS | _____ | _____ mg/l | Selenium | _____ | _____ µg/l |
| Metals, Group I: | | | Nitrogen: | | |
| Aluminum | _____ | _____ µg/l | Ammonia | _____ | _____ mg/l |
| Beryllium | _____ | _____ µg/l | Total Kjeldahl | _____ | _____ mg/l |
| Cadmium | _____ | _____ µg/l | Nitrate + Nitrite | _____ | _____ mg/l |
| Chromium | _____ | _____ µg/l | Phosphorus: | | |
| Cobalt | _____ | _____ µg/l | Total (as P) | 10/1000 | 0.12 mg/l |
| Copper | _____ | _____ µg/l | Ortho (as P) | _____ | _____ mg/l |
| Iron | _____ | _____ µg/l | pH | _____ | _____ Units |
| Lead | _____ | _____ µg/l | Phenols | _____ | _____ µg/l |
| Manganese | _____ | _____ µg/l | Residue: | | |
| Nickel | _____ | _____ µg/l | Total | _____ | _____ mg/l |
| Zinc | _____ | _____ µg/l | Suspended | _____ | _____ mg/l |
| | | | Turbidity | _____ | _____ NTU |

Laboratory reporting data ENVIRONMENTAL CHEMISTRY & MICROBIOLOGY LABORATORY

Signature Elizabeth A Bt Date 2-11-87
LABORATORY SUPERVISOR

LC-35(Rev.2/85)

State of North Carolina
Department of Natural Resources and Community Development
Division of Environmental Management
512 North Salisbury Street • Raleigh, North Carolina 27611

James G. Martin, Governor
S. Thomas Rhodes, Secretary

January 19, 1987

R. Paul Wilms
Director

Ms. Elizabeth A. Betz
Director, Natural Resources Div.
Environmental Chemistry & Microbiology Lab
Camp Lejeune, NC 28542

Dear Ms. Betz:

We have evaluated your results from the performance samples for wastewater laboratory certification received on January 9, 1987. The information from this evaluation is summarized below:

| Parameter | Value Reported | True Value | State Lab Value | Acceptable Range | Performance |
|---|---|---|---|---|---|
| *Total Phosphorus | 7.22 mg/l | 3.37 | | | Unacceptable |
| Ammonia Nitrogen | 8.23 mg/l | 8.56 | | | Acceptable |

( ) Your laboratory will be recommended for (certification) (recertification).
( ) You will be contacted to establish a date for your on-site laboratory inspection.
(*) Another sample(s) has been included for your use as required.
( ) You will be provided another sample at a future date.

Contact us at 919-733-3908 if you have questions concerning this matter.

Sincerely,

W. B. Edwards

Laboratory Section

6740/3
NREAD
8 Jan 87

Mr. W. B. Edwards
NCRD/DEM Laboratory Branch
Post Office Box 27687
Raleigh, North Carolina 27611

Dear Sir:

In accordance with your 9 December 1986 letter, the instruction and reporting form for nitrogen and phosphorus analysis is submitted. The evaluation samples were received on 15 December 1986.

Questions regarding this report should be forwarded to Ms. Elizabeth Betz, Supervisory Chemist, Natural Resources and Environmental Affairs Division, Assistant Chief of Staff, Facilities at (919) 451-5977.

Sincerely,

J. I. WOOTEN
Director

Encl:
(1) Instruction & Repting Form for Nitrogen and Phosphorus Analysis

Copy to:
ECML, NREAD (2)

Instructions and Reporting Form for
Nitrogen and Phosphorus Analysis

The contents of the enclosed ampul(s) are to be used to prepare analytical performance samples for Total Kjeldahl nitrogen and total phosphorus or ammonia nitrogen, nitrate + nitrite nitrogen, and ortho phosphorus analyses. When you are prepared to perform the analyses, open the ampul and transfer 10.0 ml from it to a one liter volumetric flask. Add distilled or deionized water to volume and mix thoroughly. See the table below for information on the concentration range and pertinent reporting information. Please report your findings in the "Value Obtained" column in accordance with the directions provided in the "Report Results" column.

Please verify that the ampul you received is NUTRIENT 4.

| Perform Analyses Marked (X) | Analysis | Concentration Range mg/l | Report Results mg/l | Value Obtained mg/l |
|---|---|---|---|---|
| | Total Kjeldahl Nitrogen | 0.1 - 10 | to 0.01 | |
| X | Total Phosphorus | 0.05 - 10 | to 0.01 | 7.22 |

Please verify that the ampul you received is NUTRIENT 2.

| Perform Analyses Marked (X) | Analysis | Concentration Range mg/l | Report Results mg/l | Value Obtained mg/l |
|---|---|---|---|---|
| | Nitrate + Nitrite Nitrogen | 0.05-10 | to 0.01 | |
| X | Ammonia Nitrogen | 0.05-10 | to 0.01 | 8.23 |
| | Ortho Phosphorus | 0.05-10 | to 0.01 | |

Return a signed copy of this completed form to:

W. B. Edwards
NRCD/DEM Laboratory Section
P. O. Box 27687
Raleigh, N. C. 27611

within 30 days of receiving this sample(s). Retain one copy for your file.

Laboratory reporting data ENVIRONMENTAL CHEMISTRY & MICROBIOLOGY LABORATORY Cert. #

Signature of Laboratory Supervisor Elizabeth A Bitz Date 22 DEC 1986

(LC-31-Rev.86)

ENCLOSURE 11

State of North Carolina
Department of Natural Resources and Community Development
Division of Environmental Management
512 North Salisbury Street • Raleigh, North Carolina 27611

James G. Martin, Governor
S. Thomas Rhodes, Secretary

R. Paul Wilms
Director

January 7, 1987

Ms. Elizabeth A. Betz
Director, Natural Resources Div.
Environmental Chemistry & Microbiology Laboratory
Camp Lejeune, NC 28542

Dear Ms. Betz:

We have evaluated results from your analysis of performance samples received on October 29, 1986. The information from this evaluation is summarized below:

| Parameter | Value Reported | True Value | State Lab Value | Acceptable Range | Performance |
|---|---|---|---|---|---|
| Oil & Grease | 37.0 mg/l | 40 mg/l | | | Satisfactory |
| Suspended Residue | 21.5 mg/l | 21 mg/l | | | " |

(X) All results are acceptable, therefore, no additional follow-up is required.
( ) Upon receipt of payment for the enclosed invoice, we will issue certification.
( ) Your laboratory will be recommended for certification.
( ) Another sample(s) has been included for your analysis as required.
( ) You will be provided another sample at a future date.

Contact us at 919-733-3908 if you have questions concerning this matter.

Sincerely,

Billy D. Byrd

Laboratory Section

cc: Ted Cashion
WiRO Supervisor

NH3 + Total P sample enclosed per telephone discussion with Ms. Betz.

W. B. Edwards

15 Dec 86

December 9, 1986

Mr. Julian I. Wooten
Director, Natural Resources Div
Environmental Chemistry &
Microbiology Laboratory
Camp Lejeune, NC 28542

Dear Mr. Wooten:

Re: North Carolina Wastewater Laboratory Certification

We have received and reviewed your December 4, 1986, letter concerning laboratory certification. We have amended our files to reflect your current laboratory name and staff listing supplied with your letter. Attached is a report for the evaluation sample analysis performed by your laboratory. As shown on the report, we have enclosed Grease & Oil and total suspended residue rerun samples for your analysis. Also enclosed are initial evaluation samples for **total phosphorus** and **ammonia nitrogen analysis.** Acceptable results are required on all parameters prior to certification.

Thank you for the information supplied. Contact us at 919-733-3908 if you have questions.

Sincerely,

WBE

W. B. Edwards
Laboratory Section

Enclosures

cc: Wilmington Regional Supervisor
Billy Byrd

Scotch® 7664 "Post-it" Routing-Request Pad

**ROUTING - REQUEST**

Please
- ☐ READ
- ☐ HANDLE
- ☐ APPROVE

and
- ☐ FORWARD
- ☐ RETURN
- ☐ KEEP OR DISCARD
- ☐ REVIEW WITH ME

To Twyla
make a copy of this for Betsy ASAP and send to lab

Date _____ From _____

## Instructions and Reporting Form for Nitrogen and Phosphorus Analysis

The contents of the enclosed ampul(s) are to be used to prepare analytical performance samples for Total Kjeldahl nitrogen and total phosphorus or ammonia nitrogen, nitrate + nitrite nitrogen, and ortho phosphorus analyses. When you are prepared to perform the analyses, open the ampul and transfer 10.0 ml from it to a one liter volumetric flask. Add distilled or deionized water to volume and mix thoroughly. See the table below for information on the concentration range and pertinent reporting information. Please report your findings in the "Value Obtained" column in accordance with the directions provided in the "Report Results" column.

Please verify that the ampul you received is NUTRIENT 4.

| Perform Analyses Marked (X) | Analysis | Concentration Range mg/l | Report Results mg/l | Value Obtained mg/l |
|---|---|---|---|---|
| | Total Kjeldahl Nitrogen | 0.1 - 10 | to 0.01 | |
| X | Total Phosphorus | 0.05 - 10 | to 0.01 | 7.22 |

Please verify that the ampul you received is NUTRIENT 2.

| Perform Analyses Marked (X) | Analysis | Concentration Range mg/l | Report Results mg/l | Value Obtained mg/l |
|---|---|---|---|---|
| | Nitrate + Nitrite Nitrogen | 0.05-10 | to 0.01 | |
| X | Ammonia Nitrogen | 0.05-10 | to 0.01 | 8.23 |
| | Ortho Phosphorus | 0.05-10 | to 0.01 | |

Return a signed copy of this completed form to:

W. B. Edwards
NRCD/DEM Laboratory Section
P. O. Box 27687
Raleigh, N. C. 27611

within 30 days of receiving this sample(s). Retain one copy for your file.

Laboratory reporting data ENVIRONMENTAL CHEMISTRY & MICROBIOLOGY LABORATORY Cert. #

Signature of Laboratory Supervisor Elizabeth C Butler Date 22 Dec 1986

ROUTING - REQUEST
READ
HANDLE
APPROVE
and
FORWARD
RETURN
KEEP OR DISCARD
REVIEW WITH ME
To [illegible]
Make a copy of this for [illegible] and send to [illegible]

(LC-31-Rev.86)

6740/3
NREAD (L)
23 Dec 86

Mr. W. B. Edwards
NCRD/DEM Laboratory Branch
Post Office Box 27687
Raleigh, North Carolina 27611

Dear Sir:

In accordance with your 9 December 1986 letter, the instruction and reporting forms for analysis of suspended residue and oil and grease are submitted.

The initial evaluation sampled for total phosphorus and ammonia nitrogen were received on 15 December 1986. Their results will follow later.

Questions regarding this report should be forwarded to Ms. Elizabeth Betz, Supervisory Chemist, Natural Resources and Environmental Affairs Division, Assistant Chief of Staff, Facilities at (919) 451-5977.

Sincerely,

D. D. SHARPE
Acting Director

Encls:
(1) Instruction & Rpting Form for Analysis for Suspended Residue
(2) Instruction & Rpting Form for Analysis of Oil & Grease

Copy to:
ECML, NREA

## Instruction and Reporting Form for Analysis of Total and Suspended Residue

Please verify that the ampul you received corresponds with the number listed below:

R

The contents of this ampul are to be used to prepare an analytical performance sample for total and suspended residue analysis.

When you are ready to perform the analysis, remove the rubber seal from the vial containing the solids, being careful not to lose particles clinging to the rubber seal.

Pour the contents of the vial through a glass funnel into the 1000 ml volumetric flask. Use deionized water to quantitatively rinse the solids from the vial and rubber seal into the volumetric flask. Rinse the glass funnel by adding deionized water to make the sample up to the 1000 ml mark.

Mix well and analyze by standard procedures.

NOTE: Some labs have found it necessary to correct for their distilled water residue content when analyzing this type of sample.

See the table below for information on the concentration range and pertinent reporting information. Please report your findings in the "Value Obtained" column in accordance with the directions provided in the "Report Results" column.

| Perform analyses Marked (X) | Analysis | Concentration Range | Report Results | Value Obtained |
|---|---|---|---|---|
| | Total Residue | 1-1000 mg/l | to 1.0 mg/l | |
| X | Suspended Residue | 1-1000 mg/l | to 1.0 mg/l | 21.5 |

Return a signed copy of this completed form to:

Dept. of Natural Resources & Community Development
Laboratory Section
Division of Environmental Management
P. O. Box 27687
Raleigh, North Carolina 27611

within 30 days of receiving this sample. Retain one copy for your file.

Laboratory Code # _____ Certificate # _____

Laboratory reporting data ENVIRONMENTAL CHEMISTRY AND MICROBIOLOGY LABORATORY

Signature of Laboratory Supervisor Elizabeth A Boy

Date Completed 15 December 1986

(LC7-4/81)

Instruction and Reporting Form for
Analysis of Oil and Grease

Please verify that the number on the ampul you received corresponds with the number listed here: G.

The contents of this ampul are to be used to prepare a sample for the analytical performance test for oil and grease analysis. Add 500 ml of distilled or deionized water and 2.5 ml concentrated HCl to a separatory funnel. Transfer 10.0 ml of the ampul contents to the separatory funnel. Complete the analysis as detailed in the required procedure. Calculate the oil or grease concentration using the formula below:

$$\text{mg/l grease or oil} = \frac{(\text{mg oil or grease in sample} - \text{mg oil or grease in blank}) \times 1000}{500}$$

(20.5 and 1.8)

Please record your result in the "Value Obtained" column in accordance with the directions provided in the "Report Results" column.

| Perform analyses Marked (X) | Analysis | Concentration R. | Report Results | Value Obtained |
|---|---|---|---|---|
| | Oil & Grease | 1-1000 mg/l | to 1 mg/l | 37.0 |

Return the ampul mailing container and a signed copy of this completed form to:

Dept. of Natural Resources & Community Development
Division of Environmental Management
Laboratory Branch
P. O. Box 27687
Raleigh, North Carolina 27611

within 30 days of receiving this sample. Retain one copy for your file.

Laboratory Code # _____ Certificate # _____

Laboratory reporting data ENVIRONMENTAL CHEMISTRY AND MICROBIOLOGY LABORATORY

Signature of laboratory supervisor Elizabeth A Betz

Date completed 15 DECEMBER 1986

(LC-33-82)

**WASTE TREATMENT LABORATORY WORKSHEET**
**MCBCL 11345/5 (REV. 11-85)**

UNKNOWN — STATE

| ☒ VERITIFED WINKLER | OVEN TEMP ° | DATE 12-15-86 |
|---|---|---|

| | | BOD ELEMENT 00310 | | | | | | SUSPENDED SOLIDS ELEMENT 00530 | | | | | | | COLIFORM | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| PLANT | | # | $DO_1$ | $DO_5$ | DEP | BOD mg/l | % | # | DISH & SOLID | DISH | SOLID | VOLUME | SOLIDS mg/l | % | DIL | TOTAL MFC/100 ML | FECAL MFC/100 ML |
| HP | R | | | | | | | UNK 27 | 1664 | 16 1449 | | | 21.5 | | | | |
| | T | | | | | | | | | | | | | | | | |
| TT | R | | | | | | | 13LK | 8978 | 16 8978 | | | | | | | |
| | T | | | | | | | | | | | | | | | | |
| CG | R | | | | | | | | | | | | | | | | |
| | T | | | | | | | | | | | | | | | | |
| CJ | R | | | | | | | | | | | | | | | | |
| | T | | | | | | | | | | | | | | | | |
| OB | R | | | | | | | | | | | | | | | | |
| | T | | | | | | | | | | | | | | | | |
| CHB | R | | | | | | | | | | | | | | | | |
| | T | | | | | | | | | | | | | | | | |
| RR | R | | | | | | | | | | | | | | | | |
| | T | | | | | | | | | | | | | | | | |
| DUP | R | | | | | | | | | | | | | | | | |
| | T | | | | | | | | | | | | | | | | |
| TD | | | | | | | | | | | | | | | | | |
| | | | | | | | | | | | | | | | | | |
| BLK | A / B | | | | | | | | | | | | | | | | |

REMARKS

1664
1449
21.5

State of North Carolina
Department of Natural Resources and Community Development
Division of Environmental Management
512 North Salisbury Street • Raleigh, North Carolina 27611

James G. Martin, Governor
S. Thomas Rhodes, Secretary

R. Paul Wilms
Director

December 9, 1986

Mr. Julian I. Wooten
Director, Natural Resources Div
Environmental Chemistry &
Microbiology Laboratory
Camp Lejeune, NC 28542

Dear Mr. Wooten:

Re: North Carolina Wastewater Laboratory Certification

We have received and reviewed your December 4, 1986, letter concerning laboratory certification. We have amended our files to reflect your current laboratory name and staff listing supplied with your letter. Attached is a report for the evaluation sample analysis performed by your laboratory. As shown on the report, we have enclosed Grease & Oil and total suspended residue rerun samples for your analysis. Also enclosed are initial evaluation samples for total phosphorus and ammonia nitrogen analysis. Acceptable results are required on all parameters prior to certification.

Thank you for the information supplied. Contact us at 919-733-3908 if you have questions.

Sincerely,

W. B. Edwards

W. B. Edwards
Laboratory Section

Enclosures

cc: Wilmington Regional Supervisor
Billy Byrd

State of North Carolina
Department of Natural Resources and Community Development
Division of Environmental Management
512 North Salisbury Street • Raleigh, North Carolina 27611

James G. Martin, Governor
S. Thomas Rhodes, Secretary

R. Paul Wilms
Director

December 9, 1986

Mr. Julian I. Wooten
Director, Natural Resources Div
Environmental Chemistry &
Microbiology Laboratory
Camp Lejeune, NC 28542

Dear Mr. Wooten:

We have evaluated your results from the performance samples for wastewater laboratory certification received on December 5, 1986 . The information from this evaluation is summarized below:

| Parameter | Value Reported | True Value | State Lab Value | Acceptable Range | Performance |
|---|---|---|---|---|---|
| BOD mg/1 | 33 | 38.65 | | 28 - 49 | Acceptable |
| *Grease & Oil mg/1 | 4.0 | 26 | | 19 - 33 | Unacceptable |
| pH Units | 4.02 | 4.01 | | 3.71-4.31 | Acceptable |
| *Suspended Residue | 3 mg/1 | 30.5 | | 22 - 39 | Unacceptable |

( ) Your laboratory will be recommended for (certification) (recertification).
( ) You will be contacted to establish a date for your on-site laboratory inspection.
(*) Another sample(s) has been included for your use as required.
( ) You will be provided another sample at a future date.

Contact us at 919-733-3908 if you have questions concerning this matter.

Sincerely,

W. B. Edwards, Jr.

Laboratory Section

cc: Wilmington Regional Supervisor
Billy Byrd
Ted Cashion

Betz

6740/3
NREAD(L)
DEC 04 1986

Mr. W. B. Edwards
NCRD/DEM, Laboratory Branch
Post Office Box 27687
Raleigh, North Carolina 27611

Dear Sir:

In accordance with your 15 October 1986 letter, the Certified Laboratory Performance Analysis Report is submitted. Your letter was received on 5 November 1986.

Since the 29 May 1985 submission of our application for Wastewater Analytical Laboratory Certification, several changes have occurred. The name of the laboratory has changed from the Water Quality Control Laboratory to the Environmental Chemistry and Microbiology Laboratory (ECML). There have been some changes in the laboratory staff and enclosed is a list of the present laboratory staff, showing their education and experience. The Base has also received their proposed National Pollutant Discharge Elimination System permits which show additional monitoring requirements for Ammonia, Total Nitrogen, Total Phosphorus, Temperature, and Dissolved Oxygen. The temperature and dissolved oxygen readings will be run by the Wastewater Treatment Plant operators under the direction of the ECML. The ammonia and total phosphorus analysis will be run by the ECML. The total nitrogen analysis is presently contracted through Applied Laboratory Services with Oxford Laboratories, Inc.

Questions regarding this report should be forwarded to Ms. Elizabeth Betz, Supervisory Chemist, Natural Resources and Environmental Affairs Division, AC/S, Facilities at (919) 451-5977.

Sincerely,

JULIAN I. WOOTEN
Director, Natural Resources Division
Assistant Chief of Staff, Facilities
By direction of the Commanding General

DEC 0 1 1808

10286

Cert No _____

## Certified Laboratory Performance Analysis Report

This form is to be used to report the results of your analysis of the enclosed performance evaluation samples. One completed copy of this form is the only report needed. Do not return the individual report forms or mailing cartons. In the appropriate space below, enter the dilution made on the ampul contents (example: 5/1000 for 5 ml to 1000 ml), the value obtained from your analysis, and return this report to:

NRCD/DEM
Laboratory Section
P. O. Box 27687
Raleigh, NC 27611-7687

within 30 days of your receiving date. Reports received after this 30 day period will be considered unsatisfactory.

| Parameter | Dilution Made | Value Obtained | | Parameter | Dilution Made | Value Obtained | |
|---|---|---|---|---|---|---|---|
| BOD | | 33 | mg/l | Metals, Group II: | | | |
| COD | | | mg/l | Antimony | | | µg/l |
| Chloride | | | mg/l | Silver | | | µg/l |
| Cyanide | | | mg/l | Thallium | | | µg/l |
| Fluoride | | | mg/l | Arsenic | | | µg/l |
| Grease & Oil | | 4.0 | mg/l | Barium | | | µg/l |
| Hardness | | | mg/l | Mercury | | | µg/l |
| MBAS | | | mg/l | Selenium | | | µg/l |
| Metals, Group I: | | | | Nitrogen: | | | |
| Aluminum | | | µg/l | Ammonia | | | mg/l |
| Beryllium | | | µg/l | Total Kjeldahl | | | mg/l |
| Cadmium | | | µg/l | Nitrate + Nitrite | | | mg/l |
| Chromium | | | µg/l | Phosphorus: | | | |
| Cobalt | | | µg/l | Total (as P) | | | mg/l |
| Copper | | | µg/l | Ortho (as P) | | | mg/l |
| Iron | | | µg/l | pH | | 4.02 | Units |
| Lead | | | µg/l | Phenols | | | µg/l |
| Manganese | | | µg/l | Residue: | | | |
| Nickel | | | µg/l | Total | | | mg/l |
| Zinc | | | µg/l | Suspended | | 3 | mg/l |
| | | | | Turbidity | | | NTU |

Laboratory reporting data ENVIRONMENTAL CHEMISTRY + MICROBIOLOGY LABORATORY

Signature Elizabeth A Bot Date 12/2/86
LABORATORY SUPERVISOR

LC-35(Rev.2/85)

ENVIRONMENTAL CHEMISTRY & MICROBIOLOGY
LABORATORY STAFF

LABORATORY DIRECTOR - Elizabeth Betz

Education: High School, B.S. in Chemistry
Certification: NCWWTPO Grade II, NCWTPO B-Well
Experience: 7 years

TECHNICIANS:

Hoy J. Burns

Education: High School, 1 year College
Certifications: Dept. of Health, Education & Welfare Clinical Laboratory Technologist, North Carolina Wastewater Operator Grade II
Experience: Navy Clinical Laboratories - 11 yrs. Navy Research Laboratories - 9 yrs., Quality Control Laboratory - 10 yrs.

Carol Shores

Education: High School, B.S., M.S.
Certifications: NCWWTPO Grade II
Experience: 2 years

Lyndia Lane

Education: High School
Experience: 6 months

Thomas H. Barbee

Education: High School, B.S. in Biology with Wildlife Option
Experience: VA Hospital in Asheville, NC - 2 yrs., ECU School of Medicine - 1 yrs., Quality Control Laboratory - 2 yrs.

State of North Carolina
Department of Natural Resources and Community Development
Division of Environmental Management
512 North Salisbury Street • Raleigh, North Carolina 27611

James G. Martin, Governor
S. Thomas Rhodes, Secretary

R. Paul Wilms
Director

October 15, 1986

Water Quality Control Laboratory
Natural Resources & Environmental Affairs Div. MC Base
Camp Lejeune, NC 28543

ATTENTION: Ms. Elizabeth A. Betz

Dear Laboratory Supervisor:

RE: Wastewater Laboratory Certification

In 1985 your laboratory submitted an application for Wastewater Laboratory Certification as required by NCAC 2H .0800. During the next few months, we will be contacting you to schedule a certification inspection of your laboratory. According to our records, your laboratory has not received and analyzed certification evaluation samples. Enclosed for your analysis are evaluation samples for parameters included in your certification application. Excluding pH, the samples are in concentrate form and must be diluted to prepare the actual sample for your analysis. Instructions are included that describe how each sample is to be diluted and reported. Within 30 days of receipt, please analyze these samples and report results to the address shown on the report form.

Contact us at 919-733-3908 if you have questions or need additional information.

Sincerely,

W. B. Edwards

W. B. Edwards
Laboratory Section

cc: Wilmington Regional Supervisor
Mr. Billy Byrd

Enclosure

## Instruction and Reporting Form for the Biochemical and Chemical Oxygen Demand Analyses

Please verify that the ampul you received corresponds with the number listed below:

D 102886.

The contents of the enclosed ampul will be used to prepare two separate samples. Both samples should be formulated using clean dry volumetric pipets to transfer the specified amount of concentrate to separate volumetric flasks. Make each to volume with good quality distilled or deionized water and proceed immediately with the analyses.

Biochemical Oxygen Demand Sample: Transfer 10.0 ml of the ampul contents to a 1 liter volumetric flask and bring to volume with distilled or deionized water. Mix the solution thoroughly to obtain representative portions for analysis. This sample must be seeded and diluted by approved procedures to obtain a proper evaluation.

Chemical Oxygen Demand Sample: Transfer 5.0 ml of the ampul contents to a 250 ml volumetric flask and bring to volume with distilled or deionized water. Mix the solution thoroughly to obtain representative portions for analysis. This sample is now ready for analysis.

See the table below for information on the concentration range and pertinent reporting information. Please report your findings in the "Value Obtained" column in accordance with the directions provided in the "Report Results" column.

| Perform Analyses Marked (X) | Analysis | Concentration Range | Report Results | Value Obtained |
|---|---|---|---|---|
| X | BOD | 10 - 200 mg/1 | to 1.0 mg/1 | 33 mg/l. |
| | COD | 1.0 - 200 mg/1 | to 1.0 mg/1 | |

Please return a signed copy of this completed form to:

Dept. of Natural Resources & Community Development
Laboratory Branch
Division of Environmental Management
P. O. Box 27687
Raleigh, North Carolina 27611-7687

within 30 days of receiving this sample. Retain one copy for your file.

Laboratory Code _____ Certificate # _____

Laboratory reporting data _____

Signature of laboratory supervisor _____

Date Completed 1 Dec 1986

LC40-8/84

Instruction and Reporting Form for
Analysis of Oil and Grease

Please verify that the number on the ampul you received corresponds with the number listed here: G 10 28 86. 10 mL/500 vol.

The contents of this ampul are to be used to prepare a sample for the analytical performance test for oil and grease analysis. Add 500 ml of distilled or deionized water and 2.5 ml concentrated HCl to a separatory funnel. Transfer ~~5.0~~ 10 ml of the ampul contents to the separatory funnel. Complete the analysis as detailed in the required procedure. Calculate the oil or grease concentration using the formula below:

$$\text{mg/l grease or oil} = \frac{(\text{mg oil or grease in sample} - \text{mg oil or grease in blank}) \times 1000}{500}$$

Please record your result in the "Value Obtained" column in accordance with the directions provided in the "Report Results" column.

| Perform analyses Marked (X) | Analysis | Concentration Range | Report Results | Value Obtained |
|---|---|---|---|---|
| X | Oil & Grease | 1-1000 mg/l | to 1 mg/l | 4.0 |

Return the ampul mailing container and a signed copy of this completed form to:

Dept. of Natural Resources & Community Development
Division of Environmental Management
Laboratory Branch
P. O. Box 27687
Raleigh, North Carolina 27611

within 30 days of receiving this sample. Retain one copy for your file.

Laboratory Code #_____ Certificate #_____

Laboratory reporting data_____

Signature of laboratory supervisor_____

Date completed 1 Dec 1986

(LC-33-82)

Instruction and Reporting Form for
Analysis of Total and Suspended Residue

Please verify that the ampul you received corresponds with the number listed below:

R 10286

The contents of this ampul are to be used to prepare an analytical performance sample for total and suspended residue analysis.

When you are ready to perform the analysis, remove the rubber seal from the vial containing the solids, being careful not to lose particles clinging to the rubber seal.

Pour the contents of the vial through a glass funnel into the 1000 ml volumetric flask. Use deionized water to quantitatively rinse the solids from the vial and rubber seal into the volumetric flask. Rinse the glass funnel by adding deionized water to make the sample up to the 1000 ml mark.

Mix well and analyze by standard procedures.

NOTE: Some labs have found it necessary to correct for their distilled water residue content when analyzing this type of sample.

See the table below for information on the concentration range and pertinent reporting information. Please report your findings in the "Value Obtained" column in accordance with the directions provided in the "Report Results" column.

| Perform analyses Marked (X) | Analysis | Concentration Range | Report Results | Value Obtained |
|---|---|---|---|---|
| | Total Residue | 1-1000 mg/l | to 1.0 mg/l | |
| X | Suspended Residue | 1-1000 mg/l | to 1.0 mg/l | 3.0 |

Return a signed copy of this completed form to:

Dept. of Natural Resources & Community Development
Laboratory Section
Division of Environmental Management
P. O. Box 27687
Raleigh, North Carolina 27611

within 30 days of receiving this sample. Retain one copy for your file.

Laboratory Code # _____ Certificate # _____

Laboratory reporting data _____

Signature of Laboratory Supervisor _____

Date Completed 2 DEC 1986

(LC7-4/81)

Instruction and Reporting Form for
Analysis of pH

Please verify that the number on the ampul you received corresponds with the number listed here: pH 10 28 86

To begin the analysis, open the ampul by snapping the top off at the narrow part of the neck and pour the contents directly into a 50 ml beaker. DO NOT DILUTE THIS CONCENTRATE. The sample is now ready for analysis.

Please record your result in the "Value Obtained" column in accordance with the directions provided in the "Report Results" column.

| Analysis | Range | Report Results | Value Obtained |
|---|---|---|---|
| pH | 0 - 14 Units | To 0.01 Units | 4.02 |

Return a signed copy of this completed form to:

W. B. Edwards, Jr.
NRCD/DEM
Laboratory Section
P. O. Box 27687
Raleigh, NC 27611-7687

within 30 days of receiving this sample. One copy for your file may be retained.

Certificate # _____

Laboratory reporting data _____

Signature of laboratory supervisor _____

Date completed 2 Dec 1986

LC-15 (Rev.5-86)

6240
NREAD
29 May 1985

Mr. W. B. Edwards, Jr.
NCRD/DEM, Laboratory Branch
Post Office Box 27687
Raleigh, North Carolina 27611

Dear Sir:

The enclosed application for Wastewater Analytical Laboratory Certification is submitted in accordance with your letter of April 5, 1985. Our present National Pollutant Discharge Elimination System permit number is NC0003239. It is our understanding that your agency has assigned a separate number to each of our seven Sewage Treatment Plants. This application satisfies requirements applicable to the Camp Geiger Sewage Treatment Plant (NC0003239); Tarawa Terrace Sewage Treatment Plant (NC0020923), and Hadnot Point Sewage Treatment Plant (NC0020877).

Except as shown in Section VII 2 of the enclosed form, all laboratory analyses are performed by the Water Quality Control Laboratory, Natural Resources and Environmental Affairs Division, Marine Corps Base, Camp Lejeune. If additional information is desired, please contact Ms. Elizabeth A. Betz, Supervisory Chemist at (919) 451-5977.

Sincerely,

J. I. WOOTEN
Director

Blind copy to:
SupvChem

Writer: D. Sharpe, NREAD 5003
Typist: J. Cross 29May85

Date 29 May 1985

STATE OF NORTH CAROLINA
DEPARTMENT OF NATURAL RESOURCES & COMMUNITY DEVELOPMENT
DIVISION OF ENVIRONMENTAL MANAGEMENT

# APPLICATION FOR WASTEWATER ANALYTICAL LABORATORY CERTIFICATION

I. Name of Laboratory[1] Water Quality Control Laboratory

Mailing Address Natural Resources & Environmental Affairs Div., MCBase
Street or Box No.
Camp Lejeune, NC 28542
City State Zip

Location 65 Molly Pitcher Drive, Hadnot Point Area
Street or Box No.
Camp Lejeune North Carolina 28542
City State Zip

Telephone Number (919)-451-5977

II. Laboratory Personnel

A. Laboratory Supervisor Elizabeth A. Betz, Supervisory Chemist

1. Education - Give your complete education history below:

Penncrest High School Media, PA 19063 Mo. June Yr. 1975
High School Name Location Date Graduated

| Education Beyond High School | Name & Location | Credits (Sem. or Qtr.Hrs) | Last Yr. Attended | Degree or Diploma & Yr. Rec'd | Major Subject |
|---|---|---|---|---|---|
| College or University | High Point College | Quantity sufficient for Degree | 1979 | BS | Chemistry |
| Graduate or Professional | | | | | |
| Other Education, Internship, etc. | | | | | |

List fields of work for which you are licensed, registered, or certified, giving date(s) and sources(s) of issuance. Wastewater Operator, Crade II

N. C. Water Treatment Operator B-Well

[1] If this is a wastewater treatment plant (WWTP) with no onsite laboratory, enter the WWTP name and address and complete sections VII and VIII only.

2. Experience:

(a) Title of present position Supervisory Chemist

Date employed Nov 1979 Full-Time X Years 5 Months 6

No. employees supervised by you 4 Part-Time ____ Years ____ Months ____

(If part-time no. hours per week ____)

Employer Camp Lejeune MCBase Address Camp Lejeune, NC 28542

Duties Supervise laboratory; responsible for monitoring Water Treatment, Wastewater Treatment and Hazardous Waste.

(b) Title of next to last position N/A

Date employed ____ Full-Time ____ Years ____ Months ____

Date Separated ____ Part-Time ____ Years ____ Months ____

No. employees supervised by you ____ (If part-time no. hours per week ____)

Name and title of supervisor ____

Employer ____ Address ____

Duties ____

(c) Title of next position N/A

Date employed ____ Full-Time ____ Years ____ Months ____

Date Separated ____ Part-Time ____ Years ____ Months ____

No. employees supervised by you ____ (If part-time no. hours per week ____)

Name and title of superviser ____

Employer ____ Address ____

Duties ____

3. References - persons familiar with your professional competency:

(a) Danny Sharpe, Supervisory Ecologist, PO Box 743, Richlands, NC 28

(b) Julian Wooten, Dir, NREAD, Marine Corps Base, Camp Lejeune, NC 28

(c) Don Beesley, LabCert. Evaluator, DHS, PO Box 28047, Raleigh, NC 2

3. Other Laboratory Personnel

| Name | Education | Years of Analytical Laboratory Experience |
|---|---|---|
| See attached sheet | | |

B. Other Laboratory Personnel

Hoy J. Burns
Education: High School, 1 year College
Certifications: Dept. of Health, Education & Welfare Clinical Laboratory Technologist, North Carolina Wastewater Operator Grade II
Experience: Navy Clinical Laboratories - 11 yrs. Navy Research Laboratories - 9 yrs. Quality Control Laboratory - 9 yrs.

Robert J. Lachapelle, 2 yrs. College, Navy Medical Technologist School
Certifications: Certified Medical Technologist
Experience: Navy Laboratories - 17 yrs. Quality Control Laboratory - 4 yrs

Gaines B. Huneycutt, Jr.
Education: High School, AAS in Fish & Wildlife Management
Certifications: North Carolina Water Treatment Operator Grade C-Well
Experience: N.C. Department of NatRes & ComDev- 6.5 yrs. Contracts & Engineers Services - 1 yr; Quality Control Laboratory - 4 yrs.

Thomas H. Barbee
Education: High School, BS in Biology with Wildlife Option
Experience: VA Hospital in Asheville, NC - 2 yrs; ECU School of Medicine - 1 yr. Quality Control Laboratory - 1 yr.

III. Parameters for which certification may be requested: Parameters presently identified in our Permit
Laboratories need to obtain certification only for those parameters which will be reported to the state in compliance with monitoring and pretreatment regulations. Please (1) circle your choice of analytical method in each instance from the list to the right of the parameter, and (2) cite your usual minimum reporting concentration for it to the far right.

| Analytical Parameter | Reference: 1979 EPA Methods | Reference: 15th Ed. Standard Methods | Reference: Others Include Reference & Methods | Min. Report Concentration (incl. Units) |
|---|---|---|---|---|
| BOD | 405.1 | (507) | | 1 mg/L |
| COD | 410.1 | 508A | | |
| | 410.2 | | | |
| | 410.3 | | | |
| | 410.4 | | | |
| Chloride | 325.3 | 407A | | |
| | 325.2 | 407B | | |
| | 325.1 | 407D | | |
| Coliform, Fecal MF | 124 (1) | (909C) | | 1/100 ML |
| Coliform, Total MF | 108 (1) | (909A) | | 1/100 ML |
| Coliform, Fecal Tube | 132 (1) | (908C) | | <4/100 ML |
| Coliform, Total Tube | 114 (1) | (908A) | | <4/100 ML |
| Cyanide | 335.2 | 412B | | |
| | 335.3 | 412C | | |
| | | 412D | | |
| Fluoride | 340.2 | 413A | | |
| | 340.1 | 413B | | |
| | 340.3 | 413C | | |
| | | 413E | | |
| Grease and Oil | 413.1 | (503A) | | 1 mg/L |
| Hardness, Total | 130.1 | 314B | | |
| | 130.2 | 314A | | |
| | 215.1+ 242.1 | | | |
| | | | | |
| MBAS | 425.1 | 512A | | |

(1) Coliform procedures taken from "Microbiological Methods for Monitoring the Environment", 1978, EPA-600/8-78-017.

| Analytical Parameter | Reference: 1979 EPA Methods | Reference: 15th Ed. Standard Methods | Reference: Others Include Reference & Methods | Min. Report Concentration (incl. Units) |
|---|---|---|---|---|
| Metals Group I | | | | |
| Aluminum | 202.1 | 303C | | |
| | 202.2 | 304 | | |
| | | 306B | | |
| Beryllium | 210.1 | 303C | | |
| | 210.2 | 304 | | |
| | | 309B | | |
| Cadmium | 213.1 | 303A | | |
| | 213.2 | 303B | | |
| | | 304 | | |
| | | 310B | | |
| Chromium, Total | 218.3 | 303A | | |
| | 218.1 | 303B | | |
| | 218.2 | 304 | | |
| | | 312A | | |
| Cobalt | 219.1 | 303A | | |
| | 219.2 | 303B | | |
| | | 304 | | |
| Copper | 220.1 | 303A | | |
| | 220.2 | 303B | | |
| | | 313B | | |
| Iron | 236.1 | 303A | | |
| | 236.2 | 303B | | |
| | | 304 | | |
| | | 315B | | |
| Lead | 239.1 | 303A | | |
| | 239.2 | 303B | | |
| | | 304 | | |
| | | 316B | | |
| Manganese | 243.1 | 303A | | |
| | 243.2 | 303B | | |
| | | 304 | | |
| | | 319B | | |

| Analytical Parameter | Reference: 1979 EPA Methods | Reference: 15th Ed. Standard Methods | Reference: Others Include Reference & Methods | Min. Report Concentration (incl. Units) |
|---|---|---|---|---|
| Nickel | 249.1 | 303A | | |
| | 249.2 | 303B | | |
| | | 304 | | |
| | | 321B | | |
| Zinc | 289.1 | 303A | | |
| | 289.2 | 303B | | |
| | | 304 | | |
| Metals Group II | | | | |
| Antimony | 204.1 | 303A | | |
| | 204.2 | 304 | | |
| Silver | 272.1 | 303A | | |
| | 272.2 | 303B | | |
| | | 304 | | |
| Thallium | 279.1 | 303A | | |
| | 279.2 | 304 | | |
| Arsenic | 206.5 | 303E | | |
| | 206.3 | 304 | | |
| | 206.2 | 307B | | |
| | 206.4 | | | |
| Barium | 208.1 | 303C | | |
| | 208.2 | 304 | | |
| Mercury | 245.1 | 303F | | |
| | 245.2 | | | |
| Selenium | 270.2 | 304 | | |
| | 270.3 | 303E | | |
| Ammonia, Nitrogen | 350.2 | 417A | | |
| | 350.3 | 417B | | |
| | 350.1 | 417D | | |
| | | 417F | | |
| Total Kjeldahl Nitrogen | 351.3 | 420A | | |
| (TKN) | 351.1 | 420B | | |
| | 351.2 | 417D | | |
| | 351.4 | 417B | | |
| | | 417E | | |

| Analytical Parameter | Reference: 1979 EPA Methods | Reference: 15th Ed. Standard Methods | Reference: Others Include Reference & Methods | Min. Report Concentration (incl. Units) |
|---|---|---|---|---|
| Nitrate plus Nitrite | 353.3 | 418C | | |
| Nitrogen | 353.2 | 418F | | |
| | 353.1 | | | |
| Total Phosphorus | 365.2 | 424C (111) | | |
| | 365.3 | 424F | | |
| | 365.1 | 424G | | |
| | 365.4 | | | |
| Orthophosphate | 365.1 | 424G | | |
| | 365.2 | 424F | | |
| | 365.3 | | | |
| pH | 150.1 | 423 | | 0.1 Unit |
| Phenols | 420.1 | 510A(2) | | |
| | | 510B(2) | | |
| | 420.2 | 510C(2) | | |
| Residue, Total | 160.3 | 209A | | |
| Residue, Suspended | 160.2 | 209D | | 1 mg/L |
| Turbidity | 180.1 | 214A | | |

(2) Standard Methods, 14th Edition

IV. Laboratory Test Equipment

| Instrument | Manufacturer/Model | Used in Analysis For: |
|---|---|---|
| pH/Dissolved Oxygen Meter | Orion 611 | Dissolved Oxygen pH |
| Oven | Fisher Model 215F | Solids |
| Coliform Incubator Bath | GCA/Precision Scientific | Fecal Coliform MF |
| Dry Air Incubator | Elconap | Total Coliform MF |
| Incubator | Precision Model 815 | BOD |
| Autoclave | Market Forge Sterilmatic | Sterilizing Micro Equipment |
| Dry Air Incubator | Precision Model 2 | Total & Fecal Coli MF |
| Oven | Fisher Model 438F | Oil & Grease |
| Water Bath | Precision | Oil & Grease |
| Shaker | Kraft Model S-500 | Oil & Grease |
| Analytical Balance | Mettler H33 | Solids & Oil & Grease |
| Vacuum Pump | Precision | Solids, Coliform |
| | | |
| | | |

(Use additional space if required)

V. Quality Control Program

1. Does your laboratory have a written quality assurance document? Yes (X) No ( ).

2. Does your laboratory analyze duplicate samples daily for each parameter? Yes (X) No ( ).

3. Does your laboratory analyze known standards for each parameter each day samples are analyzed? Yes ( ) No (X).

4. Does your laboratory meet the requirements of Table II - Required Containers, Preservation Techniques, and Holding Times as shown in the October 26, 1984, Federal Register? Yes (X) No ( ). If no, enter any deviations.

5. Briefly summarize your analytical quality assurance procedures for the parameters which certification has been requested. Use additional pages if necessary.

See attached pages

VI. Laboratory Type

1. Is your laboratory applying for certification as a commercial laboratory? Yes ( ) No (X)

2. Is your laboratory applying for certification as a municipal or industrial laboratory? Yes (X) No ( ).

3. Is your laboratory performing wastewater analysis for clients in North Carolina? Yes (X) No ( )

4. Does your laboratory wish to seek clients in North Carolina? Yes ( ) No (X).

VII. Municipal and Industrial Wastewater Treatment Plants Only

1. Enter your NPDES Permit Number NC0003239; NC0020877 and NC0020923

2. Are state required monitoring analyses for this facility performed by another laboratory? Yes ( ) No (X).
If yes, give the name and address of the laboratory and the parameters performed by that laboratory.

NOTE: Plant personnel measure or test for flow and pH.
Remaining analyses are performed by Water Quality Control Laboratory.

VIII. This statement certifies that the information contained in this application is truthful and accurate to the best of my knowledge.

Date 29 May 1985

Signature of Applicant
ELIZABETH A. BETZ
Supervisory Chemist
Title

To apply for certification, return two copies of this application to:

W. B. Edwards, Jr.
NRCD/DEM, Laboratory Branch
P. O. Box 27687
Raleigh, N. C. 27611

FOR WATER QUALITY CONTROL LABORATORY, MARINE CORPS BASE, CAMP LEJEUNE NORTH CAROLINA:

V. 5. Analytical Quality Assurance Procedures

BOD (Biochemical Oxygen Demand)

Normally the BOD samples received by this laboratory are unchlorinated. The samples are screened for chlorine before processing. Seeding is, therefore, not usually required. The dissolved oxygen meter is calibrated against a Winkler Titration with each batch of samples. Dilution water blanks are set up with each batch of dilution water. When blanks have depletions greater than 0.2 mg/l, the corresponding batch of samples is discarded. A set of glucose-glumatic acid standards, with seeding, is set up weekly. Duplication is done daily on one set of samples.

Coliform, Total & Fecal, MF & MPN

Incubator temperatures are read daily. Negative (Pre and Post) and positive controls are run with each set of samples. Any samples but potable water are run with at least two dilutions. Duplicates are also run daily. The laboratory maintains N.C. Microbiology certification for drinking water and all applicable quality control on media, sterilization of equipment, etc.

Oil and Grease

A standard and blank are run with each batch of samples

pH

Meters are standardized using low and high buffers daily. Monthly, meters are checked against certified buffers.

Residue, Solids

Oven temperatures are recorded on Laboratory Worksheets. A duplicate is analyzed daily.

General

All balances are zeroed daily. Analytical balance is checked against a set of standard weights monthly. All chemicals are analytical reagent grade. All reagents are dated when prepared. Copies of the 1979 EPA Methods and 15th Edition of Standard Methods are maintained in the Laboratory.

Date _____

STATE OF NORTH CAROLINA
DEPARTMENT OF NATURAL RESOURCES & COMMUNITY DEVELOPMENT
DIVISION OF ENVIRONMENTAL MANAGEMENT

APPLICATION
FOR
WASTEWATER ANALYTICAL LABORATORY CERTIFICATION

I. Name of Laboratory[1] WATER QUALITY CONTROL LABORATORY

Mailing Address NATURAL RESOURCES and ENVIRONMENTAL AFFAIRS DIVISION, MARINE CORPS BASE
Street or Box No.
CAMP LEJEUNE NC 28542
City State Zip

Location BLDG 65, MOLLY PITCHER DRIVE, HADNOT POINT AREA
Street or Box No.
CAMP LEJEUNE NC 28542
City State Zip

Telephone Number 919-451-5977

II. Laboratory Personnel

A. Laboratory Supervisor ELIZABETH A. BETZ, SUPERVISORY CHEMIST

1. Education - Give your complete education history below:

PENNCREST HIGH SCHOOL, MEDIA PENNSYLVANIA 19063 Mo. JUNE Yr. 1975
High School Name Location Date Graduated

| Education Beyond High School | Name & Location | Credits (Sem. or Qtr.Hrs) | Last Yr. Attended | Degree or Diploma & Yr. Rec'd | Major Subject |
|---|---|---|---|---|---|
| College or University | HIGH POINT COLLEGE | QUANTITY SUFFICIENT FOR DEGREE + | 1979 | BS | CHEMISTRY |
| Graduate or Professional | | | | | |
| Other Education, Internship, etc. | | | | | |

List fields of work for which you are licensed, registered, or certified, giving date(s) and sources(s) of issuance. N.C. WASTEWATER OPERATOR, GRADE II
N.C. WATER TREATMENT OPERATOR B-WELL

(1) If this is a wastewater treatment plant (WWTP) with no onsite laboratory, enter the WWTP name and address and complete sections VII and VIII only.

2. Experience:

(a) Title of present position SUPERVISORY CHEMIST

Date employed 11/79 Full-Time ✓ Years 5 Months 6

No. employees supervised by you 4 Part-Time ____ Years ____ Months ____

(If part-time no. hours per week ____)

Employer CAMP LEJEUNE MCB Address CAMP LEJEUNE, NC 2854

Duties SUPERVISE LABORATORY REPONSIBLE FOR MONITORING WATER TREATMENT, WASTEWATER TREATMENT AND HAZARDOUS WASTE,

(b) Title of next to last position N/A

Date employed ____ Full-Time ____ Years ____ Months ____

Date Separated ____ Part-Time ____ Years ____ Months ____

No. employees supervised by you ____ (If part-time no. hours per week ____)

Name and title of supervisor ____

Employer ____ Address ____

Duties ____

(c) Title of next position N/A

Date employed ____ Full-Time ____ Years ____ Months ____

Date Separated ____ Part-Time ____ Years ____ Months ____

No. employees supervised by you ____ (If part-time no. hours per week ____)

Name and title of superviser ____

Employer ____ Address ____

Duties ____

3. References - persons familiar with your professional competency:

(a) DANNY SHARPE, Supervisory Ecologist, P.O. BOX 743, Richlands NC 28574

(b) JULIAN WOOTEN, NREAD MARINE CORPS BASE CAMP LEJEUNE 28542

(c) DON BEESLEY, LAB Cert. Evaluator, DHS, P.O Box 28047 Raleigh 27611

B. Other Laboratory Personnel

| Name | Education | Years of Analytical Laboratory Experience |
|---|---|---|
| | | |
| | | |
| SEE ATTACHED SHEET | | |
| | | |
| | | |

VI. Laboratory Type

1. Is your laboratory applying for certification as a commercial laboratory? Yes ( ) No (X)

2. Is your laboratory applying for certification as a municipal or industrial laboratory? Yes (X) No ( ).

3. Is your laboratory performing wastewater analysis for clients in North Carolina? Yes (X) No ( )

4. Does your laboratory wish to seek clients in North Carolina? Yes ( ) No (X).

VII. Municipal and Industrial Wastewater Treatment Plants Only

1. Enter your NPDES Permit Number NC0003239, NC0020877, And NC 0020923
(That your agency has assigned each plant A number)

2. Are state required monitoring analyses for this facility performed by another laboratory? Yes ( ) No (X).
If yes, give the name and address of the laboratory and the parameters performed by that laboratory.

NOTE. PLANT personnel measure or TEST for flow and pH. Remaining Analyses are performed by Water Quality Control Laboratory. (All Samples Collected

VIII. This statement certifies that the information contained in this application is truthful and accurate to the best of my knowledge.

DO I SIGN?

Date _____ _____
Signature of Applicant

_____
Title

To apply for certification, return two copies of this application to:

W. B. Edwards, Jr.
NRCD/DEM; Laborato
P. O. Box 27687
Raleigh, N. C. 27

Julian, I recommend you sign cover letter and Below the Application. Should be mailed N.L.T. 30 MAY 85

LC-20-84

V. Quality Control Program

1. Does your laboratory have a written quality assurance document? Yes (X) No ( ).

2. Does your laboratory analyze duplicate or DILUTION samples daily for each parameter? Yes (X) No ( ).

3. Does your laboratory analyze known standards for each parameter each day samples are analyzed? Yes ( ) No (X).

4. Does your laboratory meet the requirements of Table II - Required Containers, Preservation Techniques, and Holding Times as shown in the October 26, 1984, Federal Register? Yes (X) No ( ). If no, enter any deviations.

5. Briefly summarize your analytical quality assurance procedures for the parameters which certification has been requested. Use additional pages if necessary.

SEE ATTACHED PAGES

VI. Laboratory Type

1. Is your laboratory applying for certification as a commercial laboratory? Yes ( ) No (X)

2. Is your laboratory applying for certification as a municipal or industrial laboratory? Yes (X) No ( ).

3. Is your laboratory performing wastewater analysis for clients in North Carolina? Yes (X) No ( )

4. Does your laboratory wish to seek clients in North Carolina? Yes ( ) No (X).

VII. Municipal and Industrial Wastewater Treatment Plants Only

1. Enter your NPDES Permit Number NC0003239, NC0020877, And NC 0020923
(That your agency has assigned each plant a number)

2. Are state required monitoring analyses for this facility performed by another laboratory? Yes ( ) No (X).
If yes, give the name and address of the laboratory and the parameters performed by that laboratory.

NOTE. Plant personnel measure or test for flow and pH. Remaining Analyses are performed by Water Quality Control Laboratory. Grab samples collected

VIII. This statement certifies that the information contained in this application is truthful and accurate to the best of my knowledge.

Do I sign?

Date_____ _____
Signature of Applicant

_____
Title

To apply for certification, return two copies of this application to:

W. B. Edwards, Jr.
NRCD/DEM, Laboratory Branch
P. O. Box 27687
Raleigh, N. C. 27611

V. Quality Control Program

1. Does your laboratory have a written quality assurance document? Yes (X) No ( ).

2. Does your laboratory analyze duplicate or DILUTION samples daily for each parameter? Yes (X) No ( ).

3. Does your laboratory analyze known standards for each parameter each day samples are analyzed? Yes ( ) No (X).

4. Does your laboratory meet the requirements of Table II - Required Containers, Preservation Techniques, and Holding Times as shown in the October 26, 1984, Federal Register? Yes (X) No ( ). If no, enter any deviations.

5. Briefly summarize your analytical quality assurance procedures for the parameters which certification has been requested. Use additional pages if necessary.

SEE ATTACHED PAGES

| Analytical Parameter | Reference: 1979 EPA Methods | Reference: 15th Ed. Standard Methods | Reference: Others Include Reference & Methods | Min. Report Concentration (incl. Units) |
|---|---|---|---|---|
| Nitrate plus Nitrite | 353.3 | 418C | | |
| Nitrogen | 353.2 | 418F | | |
| | 353.1 | | | |
| Total Phosphorus | 365.2 | 424C (111) | | |
| | 365.3 | 424F | | |
| | 365.1 | 424G | | |
| | 365.4 | | | |
| Orthophosphate | 365.1 | 424G | | |
| | 365.2 | 424F | | |
| | 365.3 | | | |
| pH | 150.1 | 423 | | 0.1 UNIT |
| Phenols | 420.1 | 510A(2) | | |
| | | 510B(2) | | |
| | 420.2 | 510C(2) | | |
| Residue, Total | 160.3 | 209A | | |
| Residue, Suspended | 160.2 | 209D | | 1 mg/L |
| Turbidity | 180.1 | 214A | | |

(2) Standard Methods, 14th Edition

IV. Laboratory Test Equipment

| Instrument | Manufacturer/Model | Used in Analysis For: |
|---|---|---|
| pH / DISSOLVED OXYGEN METER | ORION 611 | DISSOLVED OXYGEN, pH |
| OVEN | FISHER MODEL 215F | SOLIDS |
| COLIFORM INCUBATOR BATH | GCA/PRECISION SCIENTIFIC | FECAL COLIFORM MF |
| DRY AIR INCUBATOR | ELCONAP | TOTAL COLIFORM MF |
| INCUBATOR | PRECISION MODEL 815 | BOD |
| AUTOCLAVE | MARKET FORGE STERILMATIC | STERILIZING MICRO EQUIPMENT |
| DRY AIR INCUBATOR | PRECISION MODEL 6 | TOTAL + FECAL COLI MPN |
| OVEN | FISHER MODEL 438F | OIL + GREASE |
| WATER BATH | PRECISION | OIL + GREASE |
| SHAKER | KRAFT MODEL S-500 | OIL + GREASE |
| ANALYTICAL BALANCE | METTLER H33 | SOLIDS + OIL + GREASE |
| VACUUM PUMP | PRECISION | SOLIDS, COLIFORM |
| | | |
| | | |

(Use additional space if required)

| Analytical Parameter | Reference | | | Min. Report Concentration (incl. Units) |
|---|---|---|---|---|
| | 1979 EPA Methods | 15th Ed. Standard Methods | Others Include Reference & Methods | |
| Nickel | 249.1 | 303A | | |
| | 249.2 | 303B | | |
| | | 304 | | |
| | | 321B | | |
| Zinc | 289.1 | 303A | | |
| | 289.2 | 303B | | |
| | | 304 | | |
| Metals Group II | | | | |
| Antimony | 204.1 | 303A | | |
| | 204.2 | 304 | | |
| Silver | 272.1 | 303A | | |
| | 272.2 | 303B | | |
| | | 304 | | |
| Thallium | 279.1 | 303A | | |
| | 279.2 | 304 | | |
| Arsenic | 206.5 | 303E | | |
| | 206.3 | 304 | | |
| | 206.2 | 307B | | |
| | 206.4 | | | |
| Barium | 208.1 | 303C | | |
| | 208.2 | 304 | | |
| Mercury | 245.1 | 303F | | |
| | 245.2 | | | |
| Selenium | 270.2 | 304 | | |
| | 270.3 | 303E | | |
| Ammonia, Nitrogen | 350.2 | 417A | | |
| | 350.3 | 417B | | |
| | 350.1 | 417D | | |
| | | 417F | | |
| Total Kjeldahl Nitrogen | 351.3 | 420A | | |
| (TKN) | 351.1 | 420B | | |
| | 351.2 | 417D | | |
| | 351.4 | 417B | | |
| | | 417E | | |

PARAMETERS PRESENTLY IDENTIFIED IN OUR PERMIT

III. Parameters for which certification may be requested: Laboratories need to obtain certification only for those parameters which will be reported to the state in compliance with monitoring and pretreatment regulations. Please (1) circle your choice of analytical method in each instance from the list to the right of the parameter, and (2) cite your usual minimum reporting concentration for it to the far right.

| Analytical Parameter | Reference: 1979 EPA Methods | Reference: 15th Ed. Standard Methods | Reference: Others Include Reference & Methods | Min. Report Concentration (incl. Units) |
|---|---|---|---|---|
| BOD | 405.1 | 507 | | 1 mg/L |
| COD | 410.1 | 508A | | |
| | 410.2 | | | |
| | 410.3 | | | |
| | 410.4 | | | |
| Chloride | 325.3 | 407A | | |
| | 325.2 | 407B | | |
| | 325.1 | 407D | | |
| Coliform, Fecal MF | 124 (1) | 909C | | 1/100 mL |
| Coliform, Total MF | 108 (1) | 909A | | 1/100 mL |
| Coliform, Fecal Tube | 132 (1) | 908C | | <4/100 mL |
| Coliform, Total Tube | 114 (1) | 908A | | <4/100 mL |
| Cyanide | 335.2 | 412B | | |
| | 335.3 | 412C | | |
| | | 412D | | |
| Fluoride | 340.2 | 413A | | |
| | 340.1 | 413B | | |
| | 340.3 | 413C | | |
| | | 413E | | |
| Grease and Oil | 413.1 | 503A | | 1 mg/L |
| Hardness, Total | 130.1 | 314B | | |
| | 130.2 | 314A | | |
| | 215.1+ 242.1 | | | |
| | | | | |
| MBAS | 425.1 | 512A | | |

(1) Coliform procedures taken from "Microbiological Methods for Monitoring the Environment", 1978, EPA-600/8-78-017.

| Analytical Parameter | Reference | | | Min. Report Concentration (incl. Units) |
|---|---|---|---|---|
| | 1979 EPA Methods | 15th Ed. Standard Methods | Others Include Reference & Methods | |
| Metals Group I | | | | |
| Aluminum | 202.1 | 303C | | |
| | 202.2 | 304 | | |
| | | 306B | | |
| Beryllium | 210.1 | 303C | | |
| | 210.2 | 304 | | |
| | | 309B | | |
| Cadmium | 213.1 | 303A | | |
| | 213.2 | 303B | | |
| | | 304 | | |
| | | 310B | | |
| Chromium, Total | 218.3 | 303A | | |
| | 218.1 | 303B | | |
| | 218.2 | 304 | | |
| | | 312A | | |
| Cobalt | 219.1 | 303A | | |
| | 219.2 | 303B | | |
| | | 304 | | |
| Copper | 220.1 | 303A | | |
| | 220.2 | 303B | | |
| | | 313B | | |
| Iron | 236.1 | 303A | | |
| | 236.2 | 303B | | |
| | | 304 | | |
| | | 315B | | |
| Lead | 239.1 | 303A | | |
| | 239.2 | 303B | | |
| | | 304 | | |
| | | 316B | | |
| Manganese | 243.1 | 303A | | |
| | 243.2 | 303B | | |
| | | 304 | | |
| | | 319B | | |

V. 5. Analytical Quality Assurance Procedures

BOD (Biochemical Oxygen Demand)

Normally the BOD samples received by this laboratory are un-Chlorinated. The samples are screendd for chlorine before processing. Seeding is, therefore, not usually required. The dissolved oxygen meter is calibrated against a Winkler Titration with each batch of samples. Dilution water blanks are set up with each batch of dilution water. When blanks have depletions greater than 0.2 mg/l the corresponding batch of samples are discarded. A set of glucose-glutamic acid standards, with seeding, is set up weekly. Duplications is done daily on one set of samples.

Coliform, Total & Fecal, MF & MPN

Incubator temperatures are read daily. Negative (Pre & Post) and positive controls are run with each set of samples. Any samples but potable water are run with at least two dilutions. Duplicates are also run daily. The laboratory maintains N.C. Microbiology certification for drinking water and all applicable quality control on media, sterilation of equipment, etc.

Oil & Grease

A standard and blank are run with each batch of samples

pH

Meters are standardized using low and high buffers daily. Monthly, meters are checked against certified buffers.

Residue, Solids

Oven temperatures are recorded on Laboratory Worksheets. A duplicate is analyzed daily

General

All balances are zeroed daily. Analytical balance is checked against a set of standard weights monthly. All chemicals are analytical reagent grade. All reagents are dated when prepared. Copies of the 1979 EPA Methods and 15th Ed. of Standard Methods are maintained in the Laboratory.

B. Other Laboratory Personnel

Hoy J. Burns
Education: High School, 1 yr College
Certifications: Dept. of Health, Education & Welfare Clinical Laboratory Technologist, North Carolina Wastewater Operator Grade II
Experience: Navy Clinical Labortories-11 yrs; Navy Research Laboratories-9 yrs; Quality Control Laboratory-9 yrs

Robert J. Lachapeele, Jr. Lachapelle,
Education: High School, 2 yrs College, Navy Medical Technologist School
Certifications: Certified Medical Technologist
Experience: Navy Laboratories-17 yrs; Quality Control Laboratory-4 yrs

Gaines B. Huneycutt, Jr
Education: High School, AAS in Fish & Wildlife Management
Certifications: North Carolina Water Treatment Operator Grade C-Well
Experience: N.C. Dept of NatRes & ComDev-6.5 yrs; Contracts & Engineers Services-1 yr; Quality Control Laboratory-4 yrs

Thomas H. Barbee
Education: High School, BS in Biology with Wildlife Option
Experience: VA Hospital in Ashville, NC-2 yrs; ECU School of Medicine-1 yr; Quality Control Laboratory-1 yr.

6740/3
NREAD
8 Jul 87

Mr. N. B. Edwards
NCRD/DEM Laboratory Branch
Post Office Box 27687
Raleigh, North Carolina 27611

Dear Sir:

This is to report that the deviations cited in the 29 April 1987 on-site inspection report have been corrected. The on-site inspection report was received on 11 May 1987. Corrective actions were initiated immediately, and their descriptions are discussed below.

The laboratory now runs the glucose-glutamic acid check for the Bio-chemical Oxygen Demand (BOD) test daily instead of weekly. Two dilutions of each effluent BOD sample is run in addition to the daily duplication of one BOD sample. Duplicates of one sample for Fecal Coliform, Ammonia and Phosphorus are run, in addition to the daily dilutions of each sample.

As stated in our 4 December 1986 letter, the name of the laboratory has changed since our original application in 1985. The laboratory is now the Environmental Chemistry and Microbiology Laboratory.

The point of contact on this matter is Ms. Elisabeth Betz, Supervisory Chemist, Natural Resources and Environmental Affairs Division, Assistant Chief of Staff, Facilities at (919) 451-5977.

Sincerely,

JULIAN I. WOOTEN
Director, Natural Resources Division
By direction of the Commanding General

Copy to:
EC&ML, NREAD (2)

Writer/Typist Betz/Truanolu
Date Typed 8 Jul 87
Word Processor Number 6740/3

State of North Carolina
Department of Natural Resources and Community Development
Division of Environmental Management
512 North Salisbury Street • Raleigh, North Carolina 27611

James G. Martin, Governor
S. Thomas Rhodes, Secretary

R. Paul Wilms
Director

May 7, 1987

Ms. Elizabeth A. Betz
Camp Lejeune Water Quality Control Lab
Natural Resources & Env Affairs Div, MC Base
Camp Lejeune, NC 28542

Dear Ms. Betz:

RE: Initial Laboratory Certification Inspection

Enclosed is a copy of the referenced inspection report as prepared by Mr. Byrd. Each of the deviations must be corrected prior to certification. Within 60 days, please supply this office with a written item for item description of how these deviations were corrected. As a certification requirement, your laboratory must continue to carry out the quality controls set forth in our quality assurance guidance.

Also enclosed for your use is an updated copy of the certification regulation. In this copy, Section .0805(a)(1) has been changed to reference the October 26, 1984 Federal Register.

Thank you for your cooperation during the inspection. Contact us at 919-733-3908 if you have questions or need additional information.

Sincerely,

W. B. Edwards Jr.

William B. Edwards
Laboratory Section

Enclosures

cc: Wilmington Regional Supervisor
Mr. Billy Byrd

# ON-SITE INSPECTION REPORT

**Laboratory Name:** Camp Lejeune Water Quality Control Laboratory

**Address:** Natural Resources & Environmental Affairs Div., MC Base
Camp Lejeune, NC 28542

**Date of Inspection:** April 29, 1987 **Type: (X) Initial ( ) Maintenance**
**Evaluator:** Billy D. Byrd

**Local Person(s) contacted:** Ms. Elizabeth A. Betz

I. **Introduction- This laboratory was inspected to verify its compliance with the requirements of NCAC 2H .0800 for the analysis of wastewater samples.**

II. **General Comments:** The laboratory was in good condition including equipment, records, knowledge of tests, quality controls, and personnel.

III. **Deviations:**

BOD - The glucose-glutamic acid check was not performed daily as required.

- Only one dilution was set for each sample.

Sample duplicates - Duplicate samples were not analyzed as required.

IV. **Conclusions:** This laboratory demonstrated by the results reported in the sample performance evaluation its ability to produce quality data. After correcting the above deviations, this laboratory will meet certification requirements

**Report Prepared by:** Billy Byrd **Date:** May 7, 1987

(LC-38-84)

March 1985

NRCD/DEM Wastewater Laboratory Certification
Guidance for Preparing a Quality Assurance Document

## I. Introduction

All certified laboratories must be committed to producing quality assured data and carrying out the necessary quality controls to qualify data produced. It must be recognized that the additional controls will result in an increase in operating cost and will require additional work time. The guidance outlined here is based on the analysis of known standards to document accuracy and duplicate samples to document precision. This program also includes documentation of other standard operating procedures.

## II. Sample Receiving and Sample Identification

Each laboratory must have some system of sample identification that will keep each sample discrete. This may be an elaborate sample logging and numbering system for the larger laboratories or simply labeling the samples as influent, effluent, etc., for the smaller laboratories. Also included in this section should be instructions as to what will be done with the samples upon receipt in the laboratory. For example, samples may be preserved and stored for future analysis or they may be taken directly to the laboratory bench and analyzed.

## III. General Laboratory Practices

In order to produce quality data, the analyst must have adequate facilities, services, instrumentation, and supplies and the analyst must properly use and maintain each of these. This section should include general instructions for operating, maintaining and cleaning laboratory apparatus and equipment, and storage of chemicals.

## IV. Quality Controls

Listed below are the minimum quality controls required for North Carolina Wastewater Certification. Some laboratories are already exceeding the controls listed here and are encouraged to continue at that level. We will consider substituting existing programs that are not identical to the items listed here. For example, laboratories analyzing spiked samples could be exempted from analyzing some of the quality control standards.

### 1. BOD

a. The temperature of the BOD incubator must be maintained at 20 ± 1°C using an accurate thermometer inserted in a BOD bottle inside the incubator.

b. Check and record the incubator temperature each day.

c. Calibrate the dissolved oxygen meter each day before analyzing samples and check calibration after completing each group of analyses.

d. Samples which have a low or high pH, contain chlorine, or other toxics, must be pretreated as described in the approved procedure. After pretreatment, the samples must be seeded to provide an adequate biological population capable of oxidizing the organic material in the sample.

e. Perform a glucose-glutamic acid check each day seeded samples are analyzed.

f. Sufficient seed must be used to yield a seed correction of 0.6-1.0 mg/l.

g. Each day determine the BOD of the seed material the same as for any other unseeded sample. Calculate the seed correction from the results of the seed BOD. Do not use a seeded blank as the seed correction.

h. Analyze samples using a dilution series that will yield a dissolved oxygen usage of at least 2 mg/l or a residual of at least 1 mg/l.

i. Analyze a duplicate sample daily.

j. Perform a blank dilution water control analysis along with each batch of samples analyzed.

2. COD - Titration Procedure

a. Standardize the COD titrant each day samples are analyzed.

b. Analyze a distilled water blank with each group of samples and make proper corrections.

c. Analyze a quality control standard along with each group of samples analyzed.

Note: A 250 mg/l COD standard may be made by dissolving 0.2125 g potassium acid phthalate (that has been dried at 120°C) in one liter of distilled water. A 25 mg/l COD standard for the low level procedure may be prepared by diluting 10.0 ml of the above solution to 100 mls.

d. Analyze a duplicate sample daily.

e. Use the low level procedure for the analysis of samples with a COD of <50 mg/l.

3. COD - Colormetric

a. Prepare a standard curve as set forth in the standard procedure. As a minimum, the curve must consist of a blank and three standards (low, medium, and high).

b. In addition to the calibration standards, analyze a quality control standard each day.

c. Analyze a duplicate sample daily.

4. Coliform

a. Check the temperature of all incubators daily and maintain a log of values read.

b. The 44.5°C waterbath must be equipped with a thermometer graduated in 0.1°C increments.

c. The 35°C incubator must be equipped with a thermometer graduated in at least 0.5°C increments.

d. Log the maximum temperature and pressure of the autoclave once during each use.

e. Analyze a dilution water blank at the beginning and end of each group of samples analyzed.

f. Analyze one duplicate sample each day.

X 5. Chloride

a. Standardize the titrant each day samples are analyzed by titrating a sodium chloride standard.

b. Analyze a distilled water blank each day and make proper corrections.

c. Analyze one duplicate sample each day.

6. Hardness

a. Standardize the titrant each day samples are analyzed by titrating a calcium carbonate standard.

b. Analyze a distilled water blank each day samples are analyzed.

c. Analyze one duplicate sample daily.

X 7. Colormetric Analyses:

Cyanide, Phenol, Colormetric Fluoride, MBAS, Colormetric TKN, Colormetric Ammonia, Nitrate + Nitrite Nitrogen, Total Phosphorus, and Orthophosphate.

a. Each analyst performing the analysis must produce a standard curve consisting of at least a blank and three standards (low, medium, and high).

b. Analyze a blank and a mid-range standard along with each group of samples analyzed. If there is a significant difference in the standard analyzed and the standard curve, resolve the discrepancies or produce a new standard curve.

c. Analyze a duplicate sample with each group of samples analyzed.

X 8. Ammonia and Total Kjeldahl Nitrogen - Titration Procedure

a. Analyze a distilled water blank each day samples are analyzed.

b. For ammonia, analyze one ammonium chloride standard each day samples are analyzed.

c. For TKN, analyze one organic nitrogen standard each day samples are analyzed.

Note: A 100 mg/l organic nitrogen stock standard can be prepared by dissolving 1.0503 g of glutamic acid in 600 ml distilled water containing 1 ml concentrated $H_2SO_4$ and diluting to one liter. Diluting 10 ml of this standard and 1 ml concentrated $H_2SO_4$ to one liter with distilled water will yield a solution containing 1 mg/l of nitrogen.

d. Analyze a duplicate sample daily.

X 9. Electrode Procedure:

Fluoride, Ammonia Nitrogen, and Total Kjeldahl Nitrogen

a. Calibrate the meter according to the manufacturer's instructions.

b. Check the meter calibration by analyzing a medium level quality control standard each day.

c. Analyze a duplicate sample each day samples are analyzed.

X 10. Automated Procedures:

Ammonia Nitrogen, Total Kjeldahl Nitrogen, Nitrate + Nitrite Nitrogen, Total Phosphorus, and Orthophosphate

a. Calibrate the instrument according to the manufacturer's instructions.

b. Check the instrument calibration each day by analyzing a low, medium, and high standard.

c. Analyze a quality control standard after every ten samples and at the end of each group of analyses.

d. Analyze one duplicate sample each day samples are analyzed.

e. For TKN, analyze one organic nitrogen quality control standard each day samples are analyzed.

X 11. Oil & Grease

a. Perform a blank analysis on each batch of freon used and make proper corrections.

b. The freon must be distilled from the extraction flask using a water bath controlled at 70°C.

c. The extract must be filtered through Whatman #40 filter paper or equivalent.

d. It is recommended that a reference standard be analyzed quarterly.

X 12. pH

a. Standardize the meter using a low and high buffer daily or before each use.

b. It is recommended that a reference standard be analyzed quarterly.

c. Analyze a duplicate sample daily.

13. Total Residue and Total Suspended Residue

a. Check and record drying oven temperature each day used.

b. Analyze one duplicate sample each day samples are analyzed.

c. It is recommended that blank dishes and crucibles be carried through the entire procedure to determine if proper cooling times are being used.

d. It is recommended that a reference standard be analyzed quarterly.

14. Turbidity

a. Standards as described in the approved procedure must be secured and used.

b. Each day the turbidimeter is used, calibrate it with at least one standard for each instrument range used.

c. Analyze one duplicate sample each day samples are analyzed.

d. Samples with a turbidity of greater than 40 NTU must be diluted with turbidity-free distilled water to obtain a reading between 10 and 40 NTU. The turbidity of the original sample is then calculated using the appropriate dilution factor.

15. Metals by Flame Atomic Absorption and ICP:

Metals Group I, Metals Group II, and Barium

a. Samples must be digested as set forth in the approved procedures.

b. Calibrate the instrument each day as directed in the manufacturer's instructions.

c. Each day samples are analyzed, check the instrument calibration by analyzing a blank and a low, medium, and high standard covering the operating range of the instrument.

d. Analyze a quality control standard daily for each parameter.

e. Analyze a duplicate sample daily for each parameter.

16. Metals Hydride:

Arsenic and Selenium

a. Samples must be digested according to the approved procedures.

b. Set up the instrument according to the manufacturer's instructions.

c. Prepare a calibration curve each day by analyzing a blank and a low, medium, and high standard.

d. In addition to the calibration standards, analyze one quality control standard each day samples are analyzed.

e. Analyze one duplicate sample each day samples are analyzed.

17. Arsenic SDDC Colormetric

a. Samples must be digested according to the approved procedures.

b. Prepare a calibration curve by analyzing a blank and a low, medium, and high standard.

c. In addition to the calibration standards analyze one quality control standard each day samples are analyzed.

d. Analyze one duplicate sample daily.

18. Mercury

a. Set up the instrument according to the manufacturer's instructions and the approved procedure.

b. Prepare a calibration curve by analyzing a blank and a low, medium, and high standard each day samples are analyzed.

c. In addition to the calibration standards, analyze a quality control standard daily.

d. Analyze one duplicate sample each day samples are analyzed.

19. Atomic Absorption Furnace

Metals Group I, Metals Group II, Arsenic, Selenium, and Barium

a. Samples must be digested as set forth in the approved procedures.

b. Set up and calibrate the instrument according to the manufacturer's instructions.

c. Each day samples are analyzed, check the instrument calibration by analyzing a blank and a low, medium, and high standard covering the operating range of the instrument.

d. Analyze a quality control standard daily.

e. Analyze a duplicate sample daily.

f. Use of the method of standard additions is recommended for all samples.

20. Analytical Balance

a. The balance must be mounted on a stable surface that will allow accurate weighings of 0.1 mg.

b. Weigh a standard weight each day the balance is used and maintain a log of values read.

c. Check the balance zero before each use.

d. Check the balance with a low, medium, and high standard weight quarterly. Enter results in the balance log.

21. Approved Procedures

a. The EPA approved Federal Register procedures must be used unless a variance has been obtained from EPA.

b. A copy of the approved reference procedures must be available in the laboratory.

X 22. Chemicals, Reagents, and Glassware

a. Reagents must be prepared and used as detailed in the reference procedures.

b. Date all chemicals received and all reagent solutions prepared.

c. All chemicals should be reagent grade, when available.

d. Maintain a record of all standardizations performed.

e. We recommend that all glassware be Class A, when available.

X 23. Sample Preservation

a. Samples should be preserved immediately after collection.

b. Document the type of preservatives that are to be used and when samples are preserved.

X 24. Records

Analytical and quality control records must be available for inspection and include the following:

a. Date samples are collected and date analyzed.

b. Daily lab worksheets and workbooks.

c. Values obtained on standards, blanks, duplicate samples, and standard curves.

d. A record of all required quality controls.

e. All worksheets must contain the signature or initials of the analyst(s) performing that function.

f. All analytical records must be retained for at least three years.

25. Corrective Action

At any time that required quality controls indicate an analytical problem, reflect differences in values greater than allowed by the standard procedures, or differences in values exceed ± 25% of a known value, corrective action must be taken and corresponding samples re-analyzed if possible.

26. Statistical Control Limits

It is recommended that each laboratory calculate statistical control limits, but it is not required at this time.

a. Precisions Control Limits:

Using 30-40 sets of duplicate sample results or an annual data set, calculate precision control limits using the formulas given below:

Range (R) = 1st analysis - 2nd analysis

$$\bar{R} = \frac{\Sigma R}{n}$$

$$UWL_R = 2.51\ \bar{R}$$

$$UCL_R = 3.27\ \bar{R}$$

Where: $\bar{R}$ = average range

$UWL_R$ = Upper Warning Limit

$UCL_R$ = Upper Control Limit

2.51 = Shewhart factor for 2s (duplicate)

3.27 = Shewhart factor for 3s (duplicate)

NOTE: For procedures that have a large concentration range, the duplicate results must be grouped according to the concentration level. For example, BOD samples may be grouped as follows: 0 - 10 mg/l, 10 - 100 mg/l, and greater than 100 mg/l. Precision limits for each range would be calculated.

b. Using 30-40 results from analysis of quality control standards or an annual data set, calculate % recovery, average % recovery, standard deviation and control limits for percent recovery using these formulas:

$$P = \frac{\text{observed}}{\text{known}} \times 100$$

$$\bar{P} = \frac{\Sigma P}{n}$$

$$Sp = \sqrt{\frac{\Sigma P^2 - \frac{(\Sigma P)^2}{n}}{n-1}}$$

$$UCL_p = \bar{P} + 3\,Sp$$

$$UWL_p = \bar{P} + 2\,Sp$$

$$LCLp = \bar{P} - 3\,Sp$$

$$LWLp = \bar{P} - 2\,Sp$$

Where:

| | | |
|---|---|---|
| P | = | Percent recovery |
| $\bar{P}$ | = | Average percent recovery |
| n | = | Number of analysis |
| Sp | = | Standard deviation of percent recovery |
| UCLp | = | Upper Control limit for percent recovery |
| UWLp | = | Upper warning limit for percent recovery |
| LCLp | = | Lower control limit for percent recovery |
| LWLp | = | Lower warning limit for percent recovery |

c. Prepare Shewhart control charts for precision and accuracy.

d. Use of Control Limits

(1) These control limits can be used to determine if data is in control on a daily basis. For samples results that fall within these control limits, the established precision or accuracy assessment can be applied to the individual samples of the new sample lot.

(2) For sample results that fall outside the established control limits, the system is out of control, or the established control limits are not applicable to the new data set. Corrective action may require the sample set be analyzed again or that new control limits be established.

(3) If seven successive points fall on the same side of the $\bar{P}$ (center line) of the accuracy control charts, the system is out of control and corrective action must be taken.

e. For further information concerning statistical quality control limits, we recommend securing a copy of EPA Handbook for Analytical Quality Control in Water and Wastewater Laboratories EPA-600/4-79-019. This can be obtained by writing:

Mr. Wade Knight
Quality Assurance Officer
U.S. EPA, Region 4
College Station Rd.
Athens, GA 30613

SUBJ FILE
6700

# State of North Carolina
## Department of Natural Resources and Community Development
Division of Environmental Management
512 North Salisbury Street • Raleigh, North Carolina 27611

James G. Martin, Governor
S. Thomas Rhodes, Secretary

September 22, 1987

R. Paul Wilms
Director

Ms. Elizabeth A. Betz
NREAD, EC & M Lab
Assistant Chief of Staff, Facilities
Camp Lejeune, NC 28542

Dear Ms. Betz:

Re: Initial Wastewater Laboratory Certification

The Department of Natural Resources & Community Development, in accordance with the provisions of N.C. GS 143-215.3(a)(10), 15 NCAC 2H .0800, is pleased to certify your laboratory to perform specified water analysis required by EMC monitoring and reporting regulations 15 NCAC 2B .0500 and 2H .0900.

A certificate acknowledging the certification of your laboratory is enclosed for your use. The certificate describes the requirements and limits of your certification. Please review this certificate to insure that your laboratory is certified for all parameters required to properly meet your certification needs.

Contact us at 919-733-3908 if you have questions or need additional information.

Sincerely,

B. E. Sims, PhD
Chief, Laboratory Section

Enclosure

cc: Mr. Billy Byrd
Wilmington Regional Supervisor

*STATE OF NORTH CAROLINA DEPARTMENT OF*
*NATURAL RESOURCES & COMMUNITY DEVELOPMENT*

# Division of Environmental Management
# Laboratory Certification Program

***In accordance with the provisions of N.C.G.S. 143–215.3 (a) (1), 143–215.3 (a) (10) and NCAC 2H .0800:***

CAMP LEJEUNE ENVIRONMENTAL CHEMISTRY & MICROBIOLOGY LAB

***Is hereby certified to perform wastewater analyses (as listed on attachment I) and report monitoring data to DEM for compliance with monitoring and pretreatment regulations.***

*This certificate does not guarantee validity of data generated, but indicates the methodoloy, equipment, quality control procedures, records, and proficiency of the laboratory have been examined and found to be acceptable.*

*This certificate shall be valid until* December 31, 1989

*Certificate No.* 227

B. E. Sims, PhD
Chief, Laboratory Section

**ATTACHMENT I**
**PARAMETER CERTIFICATION**

The Camp Lejeune Environ. Chemistry & Microbiology laboratory has been evaluated and found acceptable for the measurement of the parameters listed below that are preceded by an (X).

| | | | | | |
|---|---|---|---|---|---|
| X | BOD | Metals, Group I | | | Arsenic |
| | COD | | aluminum | | Barium |
| | Chloride | | beryllium | | Mercury |
| X | Coliform, fecal MF | | cadmium | | Selenium |
| X | Coliform, total MF | | chromium, total | X | Ammonia nitrogen |
| X | Coliform, fecal tube | | cobalt | | Total Kjeldahl nitrogen |
| X | Coliform, total tube | | copper | | Nitrate + nitrite nitrogen |
| | Cyanide | | iron | X | Total phosphorus |
| | Fluoride | | lead | | Orthophosphate |
| X | Grease and oil | | manganese | X | pH |
| | Hardness, total | | nickel | | Phenols |
| | MBAS | | zinc | | Residue, total |
| | | Metals, Group II | | X | Residue, total suspended |
| | | | antimony | | Turbidity |
| | | | silver | | |
| | | | thallium | | |

This certification requires maintenance of an acceptable quality assurance program, use of approved methodology and equipment, and satisfactory performance on evaluation samples.

Laboratories may be decertified for violations as set forth in 15 NCAC 2H .0807.

Applications for certification renewal must be submitted to the State Laboratory 30 days in advance of expiration of certification.

Certificate No. 227

Effective Date September 21, 1987

Expiration Date December 31, 1989

(LC-21-Rev.85)

State of North Carolina
Department of Natural Resources and Community Development
Division of Environmental Management
512 North Salisbury Street • Raleigh, North Carolina 27611

James G. Martin, Governor
S. Thomas Rhodes, Secretary

July 14, 1987

R. Paul Wilms
Director

Ms. Elizabeth Betz
EC & ML, NREAD
Asst. Chief of Staff, Facilities
Camp Lejeune, NC 28542

Dear Ms Betz:

Your letter concerning corrective actions for your analytical procedures has been received and reviewed. The actions taken are acceptable and your laboratory meets the requirements for wastewater laboratory certification. Upon receipt of payment for the enclosed invoice, we will issue certification. We have prorated your fees for the remainder of the year. Your laboratory will be subject to a $250 maintenance fee on January 1, 1988.

Thank you again for your cooperation. Contact us at 919-733-3908 if you have questions.

Sincerely,

W. B. Edwards, Jr.

W. B. Edwards
Laboratory Section

Enclosures

cc: Mr. Billy Byrd

# INVOICE

North Carolina
Wastewater Laboratory Certification

Laboratory Name Camp Lejeune Environmental Chem. & Micro Lab Date July 15, 1987

Address ATTN: Ms. Elizabeth Betz EC & ML, NREAD, Asst. Chief of Staff, Facilities
Camp Lejeune, NC 28542

The following statement itemizes the fees required for obtaining or renewing certification in the North Carolina Wastewater Laboratory Certification Program. The fee schedule is $20.00 per analytical parameter with a minimum assessment of $250.00 per laboratory with an additional prorated charge for out-of-state travel expense included when applicable.

The assessment for your laboratory is due on August 31, 1987 and includes:

| | | |
|---|---|---|
| _____ | parameters for one year at $20 each: | $ _____ . |
| <13 | parameters minimum fee for one year: | $ _____ . |
| <13 | parameters minimum fee prorated for 4 months | $83.33 . |
| _____ | parameters prorated for _____ months: | $ _____ . |
| | Prorated out-of-state travel costs: | $ _____ . |
| | Total Assessment Due: | $83.33 . |

Please make your check payable to: NRCD/DEM Lab Certification
Mail payment to: N. C. Department Of Natural Resources & Community Development
DEM Laboratory Section
P. O. Box 27687
Raleigh, N. C. 27611

ATTENTION: W. B. Edwards, Jr.

Return one copy of this invoice with your payment.

Certifiable Analytical Parameters

| | | | |
|---|---|---|---|
| X | BOD | _____ | Arsenic |
| _____ | COD | _____ | Barium |
| _____ | Chloride | _____ | Mercury |
| X | Coliform, Fecal MF | _____ | Selenium |
| X | Coliform, Total MF | X | Ammonia Nitrogen |
| X | Coliform, Fecal Tube | _____ | Total Kjeldahl Nitrogen |
| X | Coliform, Total Tube | _____ | Nitrate plus Nitrite Nitrogen |
| _____ | Cyanide | X | Total Phosphorus |
| _____ | Fluoride | _____ | Orthophosphate |
| X | Grease and Oil | X | pH |
| _____ | Hardness, Total | _____ | Phenols |
| _____ | MBAS | _____ | Residue, Total |
| _____ | Metals, Group I | X | Residue, Total Suspended |
| _____ | Metals, Group II | _____ | Turbidity |

If there are questions concerning this billing, contact us at 919-733-3908.

State Laboratory use only

Date payment received: _____ Date Forwarded: _____
Check Number: _____ Forwarded by: _____
Forwarded to: _____

(LC-1-rev.10-85)

State of North Carolina
Department of Natural Resources and Community Development
Division of Environmental Management
512 North Salisbury Street • Raleigh, North Carolina 27611

James G. Martin, Governor
S. Thomas Rhodes, Secretary

R. Paul Wilms
Director

July 14, 1987

Ms. Elizabeth Betz
EC & ML, NREAD
Asst. Chief of Staff, Facilities
Camp Lejeune, NC 28542

Dear Ms Betz:

Your letter concerning corrective actions for your analytical procedures has been received and reviewed. The actions taken are acceptable and your laboratory meets the requirements for wastewater laboratory certification. Upon receipt of payment for the enclosed invoice, we will issue certification. We have prorated your fees for the remainder of the year. Your laboratory will be subject to a $250 maintenance fee on January 1, 1988.

Thank you again for your cooperation. Contact us at 919-733-3908 if you have questions.

Sincerely,

W. B. Edwards, Jr.

W. B. Edwards
Laboratory Section

Enclosures

cc: Mr. Billy Byrd

# INVOICE

North Carolina
Wastewater Laboratory Certification

Laboratory Name Camp Lejeune Environmental Chem. & Micro Lab Date July 15, 1987

Address ATTN: Ms. Elizabeth Betz EC & ML, NREAD, Asst. Chief of Staff, Facilities
Camp Lejeune, NC 28542

The following statement itemizes the fees required for obtaining or renewing certification in the North Carolina Wastewater Laboratory Certification Program. The fee schedule is $20.00 per analytical parameter with a minimum assessment of $250.00 per laboratory with an additional prorated charge for out-of-state travel expense included when applicable.

The assessment for your laboratory is due on August 31, 1987 and includes:

| | | |
|---|---|---|
| _____ | parameters for one year at $20 each: | $_____. |
| <13 | parameters minimum fee for one year: | $_____. |
| <13 | parameters minimum fee prorated for 4 months | $83.33. |
| _____ | parameters prorated for _____ months: | $_____. |
| | Prorated out-of-state travel costs: | $_____. |
| | Total Assessment Due: | $83.33. |

Please make your check payable to: NRCD/DEM Lab Certification
Mail payment to: N. C. Department Of Natural Resources & Community Development
DEM Laboratory Section
P. O. Box 27687
Raleigh, N. C. 27611

ATTENTION: W. B. Edwards, Jr.

Return one copy of this invoice with your payment.

## Certifiable Analytical Parameters

| | | | |
|---|---|---|---|
| X | BOD | _____ | Arsenic |
| _____ | COD | _____ | Barium |
| _____ | Chloride | _____ | Mercury |
| X | Coliform, Fecal MF | _____ | Selenium |
| X | Coliform, Total MF | X | Ammonia Nitrogen |
| X | Coliform, Fecal Tube | _____ | Total Kjeldahl Nitrogen |
| X | Coliform, Total Tube | _____ | Nitrate plus Nitrite Nitrogen |
| _____ | Cyanide | X | Total Phosphorus |
| _____ | Fluoride | _____ | Orthophosphate |
| X | Grease and Oil | X | pH |
| _____ | Hardness, Total | _____ | Phenols |
| _____ | MBAS | _____ | Residue, Total |
| _____ | Metals, Group I | X | Residue, Total Suspended |
| _____ | Metals, Group II | _____ | Turbidity |

If there are questions concerning this billing, contact us at 919-733-3908.

State Laboratory use only

Date payment received: _____ Date Forwarded: _____
Check Number: _____ Forwarded by: _____
Forwarded to: _____

(LC-1-rev.10-85)

State of North Carolina
Department of Natural Resources and Community Development
Division of Environmental Management
512 North Salisbury Street • Raleigh, North Carolina 27611

James G. Martin, Governor
S. Thomas Rhodes, Secretary

R. Paul Wilms
Director

May 7, 1987

Ms. Elizabeth A. Betz
Camp Lejeune Water Quality Control Lab
Natural Resources & Env Affairs Div, MC Base
Camp Lejeune, NC 28542

Dear Ms. Betz:

RE: Initial Laboratory Certification Inspection

Enclosed is a copy of the referenced inspection report as prepared by Mr. Byrd. Each of the deviations must be corrected prior to certification. Within 60 days, please supply this office with a written item for item description of how these deviations were corrected. As a certification requirement, your laboratory must continue to carry out the quality controls set forth in our quality assurance guidance.

Also enclosed for your use is an updated copy of the certification regulation. In this copy, Section .0805(a)(1) has been changed to reference the October 26, 1984 Federal Register.

Thank you for your cooperation during the inspection. Contact us at 919-733-3908 if you have questions or need additional information.

Sincerely,

W. B. Edwards Jr.

William B. Edwards
Laboratory Section

Enclosures

cc: Wilmington Regional Supervisor
Mr. Billy Byrd

# ON-SITE INSPECTION REPORT

**Laboratory Name:** Camp Lejeune Water Quality Control Laboratory

**Address:** Natural Resources & Environmental Affairs Div., MC Base
Camp Lejeune, NC 28542

**Date of Inspection:** April 29, 1987 **Type: (X) Initial ( ) Maintenance**
**Evaluator:** Billy D. Byrd

**Local Person(s) contacted:** Ms. Elizabeth A. Betz

I. **Introduction- This laboratory was inspected to verify its compliance with the requirements of NCAC 2H .0800 for the analysis of wastewater samples.**

II. **General Comments:** The laboratory was in good condition including equipment, records, knowledge of tests, quality controls, and personnel.

III. **Deviations:**

BOD - The glucose-glutamic acid check was not performed daily as required.

- Only one dilution was set for each sample.

Sample duplicates - Duplicate samples were not analyzed as required.

IV. **Conclusions:** This laboratory demonstrated by the results reported in the sample performance evaluation its ability to produce quality data. After correcting the above deviations, this laboratory will meet certification requirements

**Report Prepared by:** Billy Byrd **Date:** May 7, 1987

(LC-38-84)

NORTH CAROLINA ADMINISTRATIVE CODE

TITLE 15

DEPARTMENT OF NATURAL RESOURCES

AND

COMMUNITY DEVELOPMENT

CHAPTER 2

ENVIRONMENTAL MANAGEMENT DIVISION

SUBCHAPTER 2H

PROCEDURES FOR PERMITS, APPROVALS

SECTION .0800

LABORATORY CERTIFICATION



ENVIRONMENTAL MANAGEMENT COMMISSION
RALEIGH, NORTH CAROLINA

## SECTION .0800 - LABORATORY CERTIFICATION

### .0801 PURPOSE

These Regulations set forth the requirements for state certification of commercial, municipal, and industrial laboratories to perform water analyses, required by the Water and Air Quality Reporting Act, G.S. 143-215.63 et seq; Environmental Management Commission Regulations for Surface Water Monitoring, Reporting, found in Subchapter 2B of this Chapter, Section .0500 and Environmental Management Commission Regulations for Local Pretreatment Programs, found in 15 NCAC 2H .0900.

History Note: Statutory Authority G.S. 143-215.3(a)(1); 143-215.3(a)(10); Eff. February 1, 1976; Amended Eff. December 1, 1984; November 1, 1978.

### .0802 SCOPE

These Regulations apply to commercial laboratories and Class III and IV municipal or industrial wastewater treatment plant laboratories which perform water analyses for persons subject to G.S. 143-215.1, 143-215.63, et seq., or the Environmental Management Commission Regulations for Surface Water Monitoring, Reporting found in Subchapter 2B of this Chapter, Section .0500. These Regulations also apply to all wastewater treatment plant laboratories for municipalities having Local Pretreatment Programs as found in 15 NCAC 2H .0900. Municipal and industrial laboratories that perform analyses for two or less of the parameters listed in Paragraph .0804(a) of these Regulations are exempt from the requirements of these Regulations.

History Note: Statutory Authority G.S. 143-215.3(a)(1); 143-215.3(a)(10); Eff. February 1, 1976; Amended Eff. December 1, 1984.

### .0803 DEFINITIONS

The following terms as used in this Section shall have the assigned meaning:

(1) Commercial Laboratory means any laboratory which is seeking to analyze water samples for others.

(2) State means the North Carolina Division of Environmental Management of the Department of Natural Resources and Community Development, or its successor.

(3) State Laboratory means the laboratory branch of the North Carolina Division of Environmental Management, or its successor.
(4) Unacceptable results on performance evaluation samples or split samples are those that vary by more than plus or minus 25 percent of the value determined by the State Laboratory or the State Laboratory may adopt specific variance limits for a particular parameter.
(5) Certification is a declaration by the state that the personnel, equipment, records, quality control procedures, and methodology cited by the applicant are accurate and that the applicant's proficiency has been considered and found to be acceptable.
(6) Decertification is loss of certification.
(7) Recertification is reaffirmation of certification.
(8) Municipal Laboratory means a laboratory operated by a municipality or other local government to analyze samples from its wastewater treatment plant(s).
(9) Industrial Laboratory means a laboratory operated by an industry to analyze samples from its wastewater treatment plant(s).
(10) Pretreatment Program means a program of waste pretreatment requirements set up in accordance with 15 NCAC 2H .0900 and approved by the Division of Environmental Management.

History Note: Statutory Authority G.S. 143-215.3(a)(1); 143-215.3(a)(10); Eff. February 1, 1976; Amended Eff. December 1, 1984; November 1, 1978.

.0804 PARAMETERS FOR WHICH CERTIFICATION MAY BE REQUESTED

Commercial laboratories need to obtain certification only for parameters which will be reported by the client to comply with the monitoring and pretreatment regulations. Municipal and Industrial Laboratories need to obtain certification only for parameters which will be reported to the state to comply with monitoring and pretreatment regulations. A listing of selected parameters follows:

(1) BOD
(2) COD
(3) Chloride
(4) Coliform, fecal MF
(5) Coliform, total MF
(6) Coliform, fecal tube
(7) Coliform, total tube
(8) Cyanide
(9) Fluoride

(10) Grease and Oil
(11) Hardness, total
(12) MBAS
(13) Metals, Group I
  (a) aluminum
  (b) beryllium
  (c) cadmium
  (d) chromium, total
  (e) cobalt
  (f) copper
  (g) iron
  (h) lead
  (i) maganese
  (j) nickel
  (k) zinc
(14) Metals, Group II
  (a) antimony
  (b) silver
  (c) thallium
(15) Arsenic
(16) Barium
(17) Mercury
(18) Selenium
(19) Ammonia nitrogen
(20) Total Kjeldahl nitrogen (TKN)
(21) Nitrate plus nitrite nitrogen
(22) Total phosphorus
(23) Orthophosphate
(24) pH
(25) Phenols
(26) Residue, total
(27) Residue, total suspended
(28) Turbidity

History Note: Statutory Authority G.S. 143-215.3(a)(1); 143-215.3(a)(10); Eff. February 1, 1976; Amended Eff. December 1, 1984.

.0805 CERTIFICATION AND RENEWAL OF CERTIFICATION

(a) Prerequisites for Certification

(1) Laboratory Procedures. Analytical methods, sample preservation, sample containers and sample holding times shall conform to those requirements found in 40 CFR-136.3, Federal Register, Vol. 49, p. 43234 (October 26, 1984); and, Federal Register, Vol. 50, p. 690

(January 4, 1985), as submitted by the Environmental Protection Agency.

(2) Performance Evaluations. Each laboratory must demonstrate satisfactory performance on evaluation samples submitted by the State Laboratory.

(3) Supervisory Requirements.

(A) The supervisor of a commercial laboratory must have a minimum of a B.S. or A.B. degree from an accredited college or university in chemistry or closely related science curriculum plus a minimum of two years laboratory experience in analytical chemistry, or a two year associate degree from an accredited college, university, or technical institute in chemistry technology, environmental sciences, or closely related science curriculum plus a minimum of four years experience in analytical chemistry. Non-degree supervisors must have at least six years laboratory experience.

(B) The supervisor of a municipal or industrial waste water treatment plant laboratory must have a minimum of a B.S. or A.B. degree from an accredited college or university in chemistry or closely related science curriculum plus a minimum of six months laboratory experience in analytical chemistry, or a two year associate degree from an accredited college, university, or technical institute in chemistry technology, environmental sciences, or closely related science curriculum plus a minimum of two years experience in analytical chemistry. Non-degree supervisors must have at least six years laboratory experience.

(C) All laboratory supervisors are subject to review by the State Laboratory. One person may serve as supervisor of no more than two laboratories. The supervisor shall provide personal and direct supervision of the technical personnel and be held responsible for the proper performance and reporting of all analysis made for these Regulations. If the supervisor is to be absent, the supervisor shall arrange for a substitute capable of insuring the proper performance of all laboratory procedures. Existing laboratory supervisors that do not meet the requirements in this Paragraph may be accepted after review by the State Laboratory and meeting all other certification requirements.

(4) Application. Each laboratory requesting state certification or certification renewal shall submit an application in duplicate to the State Laboratory. Each application will be reviewed to determine the adequacy of personnel, equipment, records, quality control procedures, and methodology. After receiving a completed application and prior to issuing certification, a representative of the State Laboratory may visit each laboratory to verify the information in the application and the adequacy of the laboratory.

(5) Facilities and equipment. Each laboratory requesting certification must contain or be equipped with the following:

(A) A minimum of 150 sq ft of laboratory space;

(B) A minimum of 12 linear feet of laboratory bench space;

(C) A sink with hot and cold water;

(D) Adequate lighting, cooling, and heating;

(E) An analytical balance capable of weighing 0.1 mg, mounted on a heavy shock proof table;

(F) A refrigerator of adequate size that will maintain temperature of 4°C;

(G) An EPA approved or a current copy of "Standard Methods for the Analysis of Water and Wastewater" or EPAs "Methods for Chemical Analysis of Water and Wastes";

(H) A source of distilled or deionized water that will meet the minimum criteria of the approved methodologies;

(I) Glassware, chemicals, supplies, and equipment required to perform all analytical procedures included in their certification.

(6) Analytical Quality Control Program. Each laboratory shall develop and maintain a document outlining the analytical quality control practices used for the parameters included in their certification. Supporting records shall be maintained as evidence that these practices are being effectively carried out. The quality control program shall be available for inspection by the State Laboratory and include the following:

(A) All analytical quality control data pertinent to each certified analysis must be available for inspection upon request.

(B) Analyze one duplicate sample and one known standard in addition to calibration standards each

day samples are analyzed to document precision and accuracy.

(C) Any quality control procedures required by a particular approved method shall be considered as required for certification for that analysis.

(D) All quality control requirements as set forth by the State Laboratory.

(E) A corrective action policy requiring that at any time quality control results indicate an analytical problem, resolve the problem and rerun any samples involved.

(F) A policy requiring that all analytical records must be maintained for a period of three years.

(b) Issuance of Certification

(1) In the absence of substantial deficiencies, certification will be issued by the Director, Division of Environmental Management, Department of Natural Resources and Community Development, or his delegate, for each of the applicable parameters requested.

(2) Initial certifications will be issued for prorated time periods to schedule all certification renewals on the first day of January.

(3) Initial certification shall be valid for up to three years from date of issue.

(c) Maintenance of Certification.

(1) To maintain certification for each parameter, a certified laboratory must analyze up to three performance evaluation samples per year submitted by the State Laboratory as an unknown. Laboratories submitting unacceptable results on a performance evaluation sample may be required to analyze more than three samples per year.

(2) In addition, the State Laboratory may request that samples be split into two equal representative portions, one part going to the state and the other to the certified laboratory for analysis.

(3) A certified laboratory will be subject to periodic inspections during the certification period and shall make time and records available for inspections.

(4) The State Laboratory will maintain a list of certified commercial laboratories and the parameters for which the laboratories have been certified. The list will be revised every six months.

(5) The State Laboratory will maintain a list of certified municipal and industrial laboratories and the parameters for which the laboratories have been certified. The list will be revised every six months.

(6) A certified laboratory must provide the State Laboratory with written notice of laboratory supervisor changes within 30 days of such changes.

(d) Certification Renewals.

(1) Applications for certification renewal will be submitted in duplicate to the State Laboratory 30 days in advance of expiration of certification.

(2) Certification renewals of laboratories shall be issued for three years with the exception that renewals for existing certified laboratories may be prorated to make all certification renewals due on the first day of January.

(e) Discontinuation of Certification

(1) A laboratory may discontinue certification for any or all parameters by making a written request to the State Laboratory.

(2) After discontinuation of certification, a laboratory may be recertified by meeting the requirements for initial certification.

History Note: Statutory Authority G.S. 143-215.3(a)(1); 143-215.3(a)(10); Eff. February 1, 1976; Amended Eff. July 1, 1985; December 1, 1984; November 1, 1978.

.0806 FEES ASSOCIATED WITH CERTIFICATION PROGRAM

(a) Certification and Certification Renewal Fees. Before being granted certification or certification renewal, laboratories shall pay to the state a fee of twenty dollars ($20.00) for each parameter for which certification is requested, however, the minimum fee will be two hundred fifty dollars ($250.00).

(b) Certification Maintenance Fees. After certification or certification renewal has been issued certified laboratories will pay to the state a certification maintenance fee of two hundred fifty dollars ($250.00) each year. Certification maintenance fees will not be required for those years in which certification or certification renewal are required. These fees are due on or before the first day of January or the certification anniversary date.

(c) Fees may be prorated in order to make all certification renewals due on the first day of January.

(d) Out-of-state laboratories shall reimburse the state for actual travel and subsistence costs incurred in certification and maintenance of certification.

History Note: Statutory Authority G.S. 143-215.3(a)(1);
143-215.3(a)(10);
Eff. February 1, 1976;
Amended Eff. December 1, 1984.

.0807 DECERTIFICATION

(a) Laboratory Decertification. Once certified, a laboratory may lose its certification for all parameters by failing to:

(1) Maintain the facilities, or records, or personnel, or equipment, or quality control program as set forth in the application and these Regulations; or
(2) Submit truthful and accurate data reports; or
(3) Pay required fees by the date due; or
(4) Discontinue supplying data for clients or programs described in Rule .0802 of this Section during periods when a parameter decertification is in effect.

(b) Parameter Decertification. Once certified, a laboratory may loose its certification for a a parameter by failing to:

(1) Obtain acceptable results on two consecutive performance evaluation samples submitted by the State Laboratory; or
(2) Obtain acceptable results on three consecutive split samples that have also been analyzed by the State Laboratory; or
(3) Submit a split sample to the State Laboratory as requested ; or
(4) Use approved methods of analysis; or
(5) Report equipment changes that would affect its ability to perform the test within 30 days of such changes; or
(6) Report analysis of performance evaluation samples submitted by the State Laboratory within 30 days of receipt; or
(7) Maintain records and perform quality controls as set forth by these Regulations and the State Laboratory for a particular parameter; or
(8) Maintain equipment required for a particular parameter.

(c) Decertification Requirements.

(1) A decertified laboratory is not to analyze samples for the decertified parameters for programs described in Rule .0802 of this Section or clients reporting to these programs.
(2) A decertified commercial laboratory must make arrangements to supply analysis through a certified laboratory during any decertification periods or notify clients that the analysis cannot be supplied. The decertified laboratory must supply the State Laboratory

with the name of the laboratory to be used and the client(s) involved.

(3) A decertified municipal or industrial laboratory must make arrangements to have their samples analyzed by another certified laboratory during any decertification period and supply the State Laboratory with the name of the certified laboratory to be used.

History Note: Statutory Authority G.S. 143-215.3(a)(1); 143-215.3(a)(10); Eff. February 1, 1976; Amended Eff. December 1, 1984.

.0808 RECERTIFICATION

(a) A laboratory decertified because of failure to maintain sufficient or adequate facilities, or laboratory supervisor, or records, or equipment, or quality control program, or failure to pay required fees may be recertified after 30 days by showing to the satisfaction of the State Laboratory that it has corrected the deficiency(ies).

(b) A laboratory decertified for a parameter due to unacceptable results on two consecutive performance evaluation samples submitted by the State Laboratory, or on three consecutive split samples may be recertified after 60 days by reporting acceptable results on two performance evaluation samples similar to those for which approval was lost. Recertification samples may be requested at any time, however, recertification must be requested in writing at the end of the 60 day period immediately following the date of decertification.

(c) A laboratory decertified for falsified reports looses certification for all parameters and shall not be considered for any certification for a one-year period.

History Note: Statutory Authority G.S. 143-215.3(a)(1); 143-215.3(a)(10); Eff. February 1, 1976; Amended Eff. December 1, 1984.

.0809 RECIPROCITY

(a) Laboratories certified under other state certification programs may be given reciprocity certification where such programs meet the requirements of these Regulations. In requesting reciprocity certification, laboratories shall include with the application required by Regulation .0805(a) of this Section a copy of their certification and Regulation from the certifying agency.

(b) Laboratories certified on the basis of program equivalency shall pay the fees required by Regulation .0806 of this Section.

History Note: Statutory Authority G.S. 143-215.3(a)(1); 143-215.3(a)(10); Eff. February 1, 1976; Amended Eff. December 1, 1984.

.0810 ADMINISTRATION

(a) The Director of the Division of Environmental Management, Department of Natural Resources and Community Development, or his delegate, is authorized to issue certification, to reject applications for certification, to renew certification, to issue recertification, to issue decertification, and to issue reciprocity certification.

(b) Appeals. In any case where the Director of the Division of Environmental Management, Department of Natural Resources and Community Development or his delegate denies certification, or decertifies a laboratory, the laboratory may appeal to the director or his delegate for a hearing. Upon receipt of such a request, the director or his delegate shall convene a hearing of the type provided for Environmental Management Commission Regulation in 15 NCAC 2I .0300, Administrative Hearings. Appeal from the decision of a hearing officer appointed by the director or his delegate shall be governed by the Environmental Management Commission Regulation on administrative hearings.

History Note: Statutory Authority G.S. 143-215.3(a)(1); 143-215.3(a)(10); Eff. February 1, 1976; Amended Eff. December 1, 1984; November 1, 1978.

.0811 IMPLEMENTATION

(a) Commercial Laboratories

(1) Certified commercial laboratories must meet any new requirements set forth herein within 6 months of these Regulations effective date.

(2) Certification fee changes are effective January 1, 1985.

(3) Requests for new parameters can be made by submitting a proper application form.

(b) Municipal and Industrial Laboratories

(1) All Municipal and Industrial Waste Treatment Plant Laboratories subject to Rule .0802 of these Regulations are required to be certified.

(2) All Municipal and Industrial Waste Treatment Plant Laboratories subject to Rule .0802 of these Regulations

must submit an application for certification within six months of these Regulations effective date. Laboratories submitting an acceptable application will be considered in compliance with these Regulations until the State Laboratory can process the application and issue or deny certification.

(3) Laboratories that cannot meet initial certification requirements must comply with the Decertification Requirements as set forth in Rule .0807(c) of these Regulations.

History Note: Statutory Authority G.S. 143-215.3(a)(1); 143-215.3(a)(10); Eff. December 1, 1984.

March 1985

# NRCD/DEM Wastewater Laboratory Certification
# Guidance for Preparing a Quality Assurance Document

## I. Introduction

All certified laboratories must be committed to producing quality assured data and carrying out the necessary quality controls to qualify data produced. It must be recognized that the additional controls will result in an increase in operating cost and will require additional work time. The guidance outlined here is based on the analysis of known standards to document accuracy and duplicate samples to document precision. This program also includes documentation of other standard operating procedures.

## II. Sample Receiving and Sample Identification

Each laboratory must have some system of sample identification that will keep each sample discrete. This may be an elaborate sample logging and numbering system for the larger laboratories or simply labeling the samples as influent, effluent, etc., for the smaller laboratories. Also included in this section should be instructions as to what will be done with the samples upon receipt in the laboratory. For example, samples may be preserved and stored for future analysis or they may be taken directly to the laboratory bench and analyzed.

## III. General Laboratory Practices

In order to produce quality data, the analyst must have adequate facilities, services, instrumentation, and supplies and the analyst must properly use and maintain each of these. This section should include general instructions for operating, maintaining and cleaning laboratory apparatus and equipment, and storage of chemicals.

## IV. Quality Controls

Listed below are the minimum quality controls required for North Carolina Wastewater Certification. Some laboratories are already exceeding the controls listed here and are encouraged to continue at that level. We will consider substituting existing programs that are not identical to the items listed here. For example, laboratories analyzing spiked samples could be exempted from analyzing some of the quality control standards.

### 1. BOD

a. The temperature of the BOD incubator must be maintained at 20 ± 1°C using an accurate thermometer inserted in a BOD bottle inside the incubator.

b. Check and record the incubator temperature each day.

c. Calibrate the dissolved oxygen meter each day before analyzing samples and check calibration after completing each group of analyses.

d. Samples which have a low or high pH, contain chlorine, or other toxics, must be pretreated as described in the approved procedure. After pretreatment, the samples must be seeded to provide an adequate biological population capable of oxidizing the organic material in the sample.

e. Perform a glucose-glutamic acid check each day seeded samples are analyzed.

f. Sufficient seed must be used to yield a seed correction of 0.6-1.0 mg/l.

g. Each day determine the BOD of the seed material the same as for any other unseeded sample. Calculate the seed correction from the results of the seed BOD. Do not use a seeded blank as the seed correction.

h. Analyze samples using a dilution series that will yield a dissolved oxygen usage of at least 2 mg/l or a residual of at least 1 mg/l.

i. Analyze a duplicate sample daily.

j. Perform a blank dilution water control analysis along with each batch of samples analyzed.

2. COD - Titration Procedure

a. Standardize the COD titrant each day samples are analyzed.

b. Analyze a distilled water blank with each group of samples and make proper corrections.

c. Analyze a quality control standard along with each group of samples analyzed.

Note: A 250 mg/l COD standard may be made by dissolving 0.2125 g potassium acid phthalate (that has been dried at 120°C) in one liter of distilled water. A 25 mg/l COD standard for the low level procedure may be prepared by diluting 10.0 ml of the above solution to 100 mls.

d. Analyze a duplicate sample daily.

e. Use the low level procedure for the analysis of samples with a COD of <50 mg/l.

3. COD - Colormetric

a. Prepare a standard curve as set forth in the standard procedure. As a minimum, the curve must consist of a blank and three standards (low, medium, and high).

b. In addition to the calibration standards, analyze a quality control standard each day.

c. Analyze a duplicate sample daily.

4. Coliform

a. Check the temperature of all incubators daily and maintain a log of values read.

b. The 44.5°C waterbath must be equipped with a thermometer graduated in 0.1°C increments.

c. The 35°C incubator must be equipped with a thermometer graduated in at least 0.5°C increments.

d. Log the maximum temperature and pressure of the autoclave once during each use.

e. Analyze a dilution water blank at the beginning and end of each group of samples analyzed.

f. Analyze one duplicate sample each day.

5. Chloride

a. Standardize the titrant each day samples are analyzed by titrating a sodium chloride standard.

b. Analyze a distilled water blank each day and make proper corrections.

c. Analyze one duplicate sample each day.

6. Hardness

a. Standardize the titrant each day samples are analyzed by titrating a calcium carbonate standard.

b. Analyze a distilled water blank each day samples are analyzed.

c. Analyze one duplicate sample daily.

7. Colormetric Analyses:

Cyanide, Phenol, Colormetric Fluoride, MBAS, Colormetric TKN, Colormetric Ammonia, Nitrate + Nitrite Nitrogen, Total Phosphorus, and Orthophosphate.

a. Each analyst performing the analysis must produce a standard curve consisting of at least a blank and three standards (low, medium, and high).

b. Analyze a blank and a mid-range standard along with each group of samples analyzed. If there is a significant difference in the standard analyzed and the standard curve, resolve the discrepancies or produce a new standard curve.

c. Analyze a duplicate sample with each group of samples analyzed.

8. Ammonia and Total Kjeldahl Nitrogen - Titration Procedure

a. Analyze a distilled water blank each day samples are analyzed.

b. For ammonia, analyze one ammonium chloride standard each day samples are analyzed.

c. For TKN, analyze one organic nitrogen standard each day samples are analyzed.

Note: A 100 mg/l organic nitrogen stock standard can be prepared by dissolving 1.0503 g of glutamic acid in 600 ml distilled water containing 1 ml concentrated $H_2SO_4$ and diluting to one liter. Diluting 10 ml of this standard and 1 ml concentrated $H_2SO_4$ to one liter with distilled water will yield a solution containing 1 mg/l of nitrogen.

d. Analyze a duplicate sample daily.

9. Electrode Procedure:

Fluoride, Ammonia Nitrogen, and Total Kjeldahl Nitrogen

a. Calibrate the meter according to the manufacturer's instructions.

b. Check the meter calibration by analyzing a medium level quality control standard each day.

c. Analyze a duplicate sample each day samples are analyzed.

10. Automated Procedures:

Ammonia Nitrogen, Total Kjeldahl Nitrogen, Nitrate + Nitrite Nitrogen, Total Phosphorus, and Orthophosphate

a. Calibrate the instrument according to the manufacturer's instructions.

b. Check the instrument calibration each day by analyzing a low, medium, and high standard.

c. Analyze a quality control standard after every ten samples and at the end of each group of analyses.

d. Analyze one duplicate sample each day samples are analyzed.

e. For TKN, analyze one organic nitrogen quality control standard each day samples are analyzed.

11. Oil & Grease

a. Perform a blank analysis on each batch of freon used and make proper corrections.

b. The freon must be distilled from the extraction flask using a water bath controlled at $70^{\circ}C$.

c. The extract must be filtered through Whatman #40 filter paper or equivalent.

d. It is recommended that a reference standard be analyzed quarterly.

12. pH

a. Standardize the meter using a low and high buffer daily or before each use.

b. It is recommended that a reference standard be analyzed quarterly.

c. Analyze a duplicate sample daily.

13. Total Residue and Total Suspended Residue

a. Check and record drying oven temperature each day used.

b. Analyze one duplicate sample each day samples are analyzed.

c. It is recommended that blank dishes and crucibles be carried through the entire procedure to determine if proper cooling times are being used.

d. It is recommended that a reference standard be analyzed quarterly.

14. Turbidity

a. Standards as described in the approved procedure must be secured and used.

b. Each day the turbidimeter is used, calibrate it with at least one standard for each instrument range used.

c. Analyze one duplicate sample each day samples are analyzed.

d. Samples with a turbidity of greater than 40 NTU must be diluted with turbidity-free distilled water to obtain a reading between 10 and 40 NTU. The turbidity of the original sample is then calculated using the appropriate dilution factor.

15. Metals by Flame Atomic Absorption and ICP:

Metals Group I, Metals Group II, and Barium

a. Samples must be digested as set forth in the approved procedures.

b. Calibrate the instrument each day as directed in the manufacturer's instructions.

c. Each day samples are analyzed, check the instrument calibration by analyzing a blank and a low, medium, and high standard covering the operating range of the instrument.

d. Analyze a quality control standard daily for each parameter.

e. Analyze a duplicate sample daily for each parameter.

16. Metals Hydride:

Arsenic and Selenium

a. Samples must be digested according to the approved procedures.

b. Set up the instrument according to the manufacturer's instructions.

c. Prepare a calibration curve each day by analyzing a blank and a low, medium, and high standard.

d. In addition to the calibration standards, analyze one quality control standard each day samples are analyzed.

e. Analyze one duplicate sample each day samples are analyzed.

17. Arsenic SDDC Colormetric

a. Samples must be digested according to the approved procedures.

b. Prepare a calibration curve by analyzing a blank and a low, medium, and high standard.

c. In addition to the calibration standards analyze one quality control standard each day samples are analyzed.

d. Analyze one duplicate sample daily.

18. Mercury

a. Set up the instrument according to the manufacturer's instructions and the approved procedure.

b. Prepare a calibration curve by analyzing a blank and a low, medium, and high standard each day samples are analyzed.

c. In addition to the calibration standards, analyze a quality control standard daily.

d. Analyze one duplicate sample each day samples are analyzed.

19. Atomic Absorption Furnace

Metals Group I, Metals Group II, Arsenic, Selenium, and Barium

a. Samples must be digested as set forth in the approved procedures.

b. Set up and calibrate the instrument according to the manufacturer's instructions.

c. Each day samples are analyzed, check the instrument calibration by analyzing a blank and a low, medium, and high standard covering the operating range of the instrument.

d. Analyze a quality control standard daily.

e. Analyze a duplicate sample daily.

f. Use of the method of standard additions is recommended for all samples.

20. Analytical Balance

a. The balance must be mounted on a stable surface that will allow accurate weighings of 0.1 mg.

b. Weigh a standard weight each day the balance is used and maintain a log of values read.

c. Check the balance zero before each use.

d. Check the balance with a low, medium, and high standard weight quarterly. Enter results in the balance log.

21. Approved Procedures

a. The EPA approved Federal Register procedures must be used unless a variance has been obtained from EPA.

b. A copy of the approved reference procedures must be available in the laboratory.

22. Chemicals, Reagents, and Glassware

a. Reagents must be prepared and used as detailed in the reference procedures.

b. Date all chemicals received and all reagent solutions prepared.

c. All chemicals should be reagent grade, when available.

d. Maintain a record of all standardizations performed.

e. We recommend that all glassware be Class A, when available.

23. Sample Preservation

a. Samples should be preserved immediately after collection.

b. Document the type of preservatives that are to be used and when samples are preserved.

24. Records

Analytical and quality control records must be available for inspection and include the following:

a. Date samples are collected and date analyzed.

b. Daily lab worksheets and workbooks.

c. Values obtained on standards, blanks, duplicate samples, and standard curves.

d. A record of all required quality controls.

e. All worksheets must contain the signature or initials of the analyst(s) performing that function.

f. All analytical records must be retained for at least three years.

25. Corrective Action

At any time that required quality controls indicate an analytical problem, reflect differences in values greater than allowed by the standard procedures, or differences in values exceed ± 25% of a known value, corrective action must be taken and corresponding samples re-analyzed if possible.

26. Statistical Control Limits

It is recommended that each laboratory calculate statistical control limits, but it is not required at this time.

a. Precisions Control Limits:

Using 30-40 sets of duplicate sample results or an annual data set, calculate precision control limits using the formulas given below:

Range (R) = 1st analysis - 2nd analysis

$$\bar{R} = \frac{\Sigma R}{n}$$

$$UWL_R = 2.51\ \bar{R}$$

$$UCL_R = 3.27\ \bar{R}$$

Where: $\bar{R}$ = average range

$UWL_R$ = Upper Warning Limit

$UCL_R$ = Upper Control Limit

2.51 = Shewhart factor for 2s (duplicate)

3.27 = Shewhart factor for 3s (duplicate)

NOTE: For procedures that have a large concentration range, the duplicate results must be grouped according to the concentration level. For example, BOD samples may be grouped as follows: 0 - 10 mg/1, 10 - 100 mg/1, and greater than 100 mg/1. Precision limits for each range would be calculated.

b. Using 30-40 results from analysis of quality control standards or an annual data set, calculate % recovery, average % recovery, standard deviation and control limits for percent recovery using these formulas:

$$P = \frac{\text{observed}}{\text{known}} \times 100$$

$$\bar{P} = \frac{\Sigma P}{n}$$

$$Sp = \sqrt{\frac{\Sigma P^2 - \frac{(\Sigma P)^2}{n}}{n - 1}}$$

$$UCL_p = \bar{P} + 3\,Sp$$

$$UWL_p = \bar{P} + 2\,Sp$$

$$LCLp = \bar{P} - 3\,Sp$$

$$LWLp = \bar{P} - 2\,Sp$$

Where:

| | | |
|---|---|---|
| P | = | Percent recovery |
| $\bar{P}$ | = | Average percent recovery |
| n | = | Number of analysis |
| Sp | = | Standard deviation of percent recovery |
| UCLp | = | Upper Control limit for percent recovery |
| UWLp | = | Upper warning limit for percent recovery |
| LCLp | = | Lower control limit for percent recovery |
| LWLp | = | Lower warning limit for percent recovery |

c. Prepare Shewhart control charts for precision and accuracy.

d. Use of Control Limits

(1) These control limits can be used to determine if data is in control on a daily basis. For samples results that fall within these control limits, the established precision or accuracy assessment can be applied to the individual samples of the new sample lot.

(2) For sample results that fall outside the established control limits, the system is out of control, or the established control limits are not applicable to the new data set. Corrective action may require the sample set be analyzed again or that new control limits be established.

(3) If seven successive points fall on the same side of the $\bar{P}$ (center line) of the accuracy control charts, the system is out of control and corrective action must be taken.

e. For further information concerning statistical quality control limits, we recommend securing a copy of EPA Handbook for Analytical Quality Control in Water and Wastewater Laboratories EPA-600/4-79-019. This can be obtained by writing:

Mr. Wade Knight
Quality Assurance Officer
U.S. EPA, Region 4
College Station Rd.
Athens, GA 30613

File + Work Copy

Appears to contain useful Info. Please refer to Previous Correspondence for Due Dates.

D Sharpe

Development

27611

S. Thomas Rhodes, Secretary

APR 5 1985

Dear NPDES Permit Administrator:

RE: Quality Assurance Guidance and Steps Involved in Securing Certification

Enclosed is the quality assurance guidance that was promised in Mr. Helms' January 1985 letter that transmitted wastewater laboratory certification information. Part .0805(a)(6) of the certification regulation states that each laboratory shall develop and maintain a quality assurance document outlining the quality control practices of the laboratory. Laboratories are not expected to have a quality control document, but are expected to begin developing one.

Parts I - III of the enclosed information is general guidance for developing a quality control plan and part IV is a listing of the minimum controls required for certification. Although it will be July or August 1985 before we begin processing certification applications, we encourage each laboratory to begin implementing the required quality controls so they will be better prepared for certification. We will consider substituting existing programs that are not identical to the enclosed required program. For example, laboratories analyzing spiked samples could be exempted from analyzing some of the quality control standards.

Questions have been raised concerning quality controls for parameters for which standards are not readily available (Ex. Coliform). Quality control requirements for these parameters have been modified. At a future public hearing, we will propose changes in the certification regulations that properly address quality controls for these parameters.

Some laboratories have requested information about the steps involved in securing certification. The following is a summary of the steps involved.

1. The laboratory completes the application forms and submits them to the Division of Environmental Management (DEM) Laboratory.

2. DEM Laboratory reviews the applications, notes any deficiencies and returns the application for any needed information.

P.O. Box 27687, Raleigh, North Carolina 27611-7687 Telephone 919-733-4984

State of North Carolina
Department of Natural Resources and Community Development
512 North Salisbury Street • Raleigh, North Carolina 27611

James G. Martin, Governor

S. Thomas Rhodes, Secretary

Division of Environmental Management

APR 5 1985

Dear NPDES Permit Administrator:

RE: Quality Assurance Guidance and Steps Involved in Securing Certification

Enclosed is the quality assurance guidance that was promised in Mr. Helms' January 1985 letter that transmitted wastewater laboratory certification information. Part .0805(a)(6) of the certification regulation states that each laboratory shall develop and maintain a quality assurance document outlining the quality control practices of the laboratory. Laboratories are not expected to have a quality control document, but are expected to begin developing one.

Parts I - III of the enclosed information is general guidance for developing a quality control plan and part IV is a listing of the minimum controls required for certification. Although it will be July or August 1985 before we begin processing certification applications, we encourage each laboratory to begin implementing the required quality controls so they will be better prepared for certification. We will consider substituting existing programs that are not identical to the enclosed required program. For example, laboratories analyzing spiked samples could be exempted from analyzing some of the quality control standards.

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Some laboratories have requested information about the steps involved in securing certification. The following is a summary of the steps involved.

1. The laboratory completes the application forms and submits them to the Division of Environmental Management (DEM) Laboratory.

2. DEM Laboratory reviews the applications, notes any deficiencies and returns the application for any needed information.

P.O. Box 27687, Raleigh, North Carolina 27611-7687 Telephone 919-733-4984

3. DEM Laboratory mails performance evaluation samples to the laboratories seeking certification. This will begin in July or August 1985.

4. Your laboratory analyzes the samples and submits results to the DEM Laboratory.

5. DEM Laboratory reviews and evaluates these results and, if necessary, sends rerun samples. The DEM Laboratory will assist any laboratories having problems producing acceptable results. Assistance may be in the form of known samples, methodology, work forms, or quality controls.

6. The DEM Laboratory schedules and performs an onsite laboratory inspection. Time sensitive samples may be delivered during the inspection.

7. An inspection report listing any observed deficiencies will be prepared and mailed.

8. The laboratory seeking certification agrees to take any required corrective actions. Reasonable time will be given for taking corrective actions.

9. The laboratory requesting certification is billed the appropriate fees.

10. Upon receipt of payment, DEM issues certification for up to three years.

We continue to look forward to working with your laboratory in the certification program. If you have any questions concerning the laboratory certification program, contact Mr. Billy D. Byrd or Mr. William B. Edwards, Jr. at 919-733-3908.

Sincerely,

W. B. Edwards, Jr.

William B. Edwards, Jr.

Enclosure

March 1985

# NRCD/DEM Wastewater Laboratory Certification
# Guidance for Preparing a Quality Assurance Document

## I. Introduction

All certified laboratories must be committed to producing quality assured data and carrying out the necessary quality controls to qualify data produced. It must be recognized that the additional controls will result in an increase in operating cost and will require additional work time. The guidance outlined here is based on the analysis of known standards to document accuracy and duplicate samples to document precision. This program also includes documentation of other standard operating procedures.

## II. Sample Receiving and Sample Identification

Each laboratory must have some system of sample identification that will keep each sample discrete. This may be an elaborate sample logging and numbering system for the larger laboratories or simply labeling the samples as influent, effluent, etc., for the smaller laboratories. Also included in this section should be instructions as to what will be done with the samples upon receipt in the laboratory. For example, samples may be preserved and stored for future analysis or they may be taken directly to the laboratory bench and analyzed.

## III. General Laboratory Practices

In order to produce quality data, the analyst must have adequate facilities, services, instrumentation, and supplies and the analyst must properly use and maintain each of these. This section should include general instructions for operating, maintaining and cleaning laboratory apparatus and equipment, and storage of chemicals.

## IV. Quality Controls

Listed below are the minimum quality controls required for North Carolina Wastewater Certification. Some laboratories are already exceeding the controls listed here and are encouraged to continue at that level. We will consider substituting existing programs that are not identical to the items listed here. For example, laboratories analyzing spiked samples could be exempted from analyzing some of the quality control standards.

### 1. BOD

a. The temperature of the BOD incubator must be maintained at 20 ± 1°C using an accurate thermometer inserted in a BOD bottle inside the incubator.

b. Check and record the incubator temperature each day.

c. Calibrate the dissolved oxygen meter each day before analyzing samples and check calibration after completing each group of analyses.

d. Samples which have a low or high pH, contain chlorine, or other toxics, must be pretreated as described in the approved procedure. After pretreatment, the samples must be seeded to provide an adequate biological population capable of oxidizing the organic material in the sample.

e. Perform a glucose-glutamic acid check each day seeded samples are analyzed.

f. Sufficient seed must be used to yield a seed correction of 0.6-1.0 mg/l.

g. Each day determine the BOD of the seed material the same as for any other unseeded sample. Calculate the seed correction from the results of the seed BOD. Do not use a seeded blank as the seed correction.

h. Analyze samples using a dilution series that will yield a dissolved oxygen usage of at least 2 mg/l or a residual of at least 1 mg/l.

i. Analyze a duplicate sample daily.

j. Perform a blank dilution water control analysis along with each batch of samples analyzed.

2. COD - Titration Procedure

a. Standardize the COD titrant each day samples are analyzed.

b. Analyze a distilled water blank with each group of samples and make proper corrections.

c. Analyze a quality control standard along with each group of samples analyzed.

Note: A 250 mg/l COD standard may be made by dissolving 0.2125 g potassium acid phthalate (that has been dried at 120°C) in one liter of distilled water. A 25 mg/l COD standard for the low level procedure may be prepared by diluting 10.0 ml of the above solution to 100 mls.

d. Analyze a duplicate sample daily.

e. Use the low level procedure for the analysis of samples with a COD of <50 mg/l.

3. COD - Colormetric

a. Prepare a standard curve as set forth in the standard procedure. As a minimum, the curve must consist of a blank and three standards (low, medium, and high).

b. In addition to the calibration standards, analyze a quality control standard each day.

c. Analyze a duplicate sample daily.

4. Coliform

a. Check the temperature of all incubators daily and maintain a log of values read.

b. The 44.5°C waterbath must be equipped with a thermometer graduated in 0.1°C increments.

c. The 35°C incubator must be equipped with a thermometer graduated in at least 0.5°C increments.

d. Log the maximum temperature and pressure of the autoclave once during each use.

e. Analyze a dilution water blank at the beginning and end of each group of samples analyzed.

f. Analyze one duplicate sample each day.

5. Chloride

a. Standardize the titrant each day samples are analyzed by titrating a sodium chloride standard.

b. Analyze a distilled water blank each day and make proper corrections.

c. Analyze one duplicate sample each day.

6. Hardness

a. Standardize the titrant each day samples are analyzed by titrating a calcium carbonate standard.

b. Analyze a distilled water blank each day samples are analyzed.

c. Analyze one duplicate sample daily.

7. Colormetric Analyses:

Cyanide, Phenol, Colormetric Fluoride, MBAS, Colormetric TKN, Colormetric Ammonia, Nitrate + Nitrite Nitrogen, Total Phosphorus, and Orthophosphate.

a. Each analyst performing the analysis must produce a standard curve consisting of at least a blank and three standards (low, medium, and high).

b. Analyze a blank and a mid-range standard along with each group of samples analyzed. If there is a significant difference in the standard analyzed and the standard curve, resolve the discrepancies or produce a new standard curve.

c. Analyze a duplicate sample with each group of samples analyzed.

8. Ammonia and Total Kjeldahl Nitrogen - Titration Procedure

a. Analyze a distilled water blank each day samples are analyzed.

b. For ammonia, analyze one ammonium chloride standard each day samples are analyzed.

c. For TKN, analyze one organic nitrogen standard each day samples are analyzed.

Note: A 100 mg/l organic nitrogen stock standard can be prepared by dissolving 1.0503 g of glutamic acid in 600 ml distilled water containing 1 ml concentrated $H_2SO_4$ and diluting to one liter. Diluting 10 ml of this standard and 1 ml concentrated $H_2SO_4$ to one liter with distilled water will yield a solution containing 1 mg/l of nitrogen.

d. Analyze a duplicate sample daily.

9. Electrode Procedure:

Fluoride, Ammonia Nitrogen, and Total Kjeldahl Nitrogen

a. Calibrate the meter according to the manufacturer's instructions.

b. Check the meter calibration by analyzing a medium level quality control standard each day.

c. Analyze a duplicate sample each day samples are analyzed.

10. Automated Procedures:

Ammonia Nitrogen, Total Kjeldahl Nitrogen, Nitrate + Nitrite Nitrogen, Total Phosphorus, and Orthophosphate

a. Calibrate the instrument according to the manufacturer's instructions.

b. Check the instrument calibration each day by analyzing a low, medium, and high standard.

c. Analyze a quality control standard after every ten samples and at the end of each group of analyses.

d. Analyze one duplicate sample each day samples are analyzed.

e. For TKN, analyze one organic nitrogen quality control standard each day samples are analyzed.

11. Oil & Grease

a. Perform a blank analysis on each batch of freon used and make proper corrections.

b. The freon must be distilled from the extraction flask using a water bath controlled at 70°C.

c. The extract must be filtered through Whatman #40 filter paper or equivalent.

d. It is recommended that a reference standard be analyzed quarterly.

12. pH

a. Standardize the meter using a low and high buffer daily or before each use.

b. It is recommended that a reference standard be analyzed quarterly.

c. Analyze a duplicate sample daily.

13. Total Residue and Total Suspended Residue

a. Check and record drying oven temperature each day used.

b. Analyze one duplicate sample each day samples are analyzed.

c. It is recommended that blank dishes and crucibles be carried through the entire procedure to determine if proper cooling times are being used.

d. It is recommended that a reference standard be analyzed quarterly.

14. Turbidity

a. Standards as described in the approved procedure must be secured and used.

b. Each day the turbidimeter is used, calibrate it with at least one standard for each instrument range used.

c. Analyze one duplicate sample each day samples are analyzed.

d. Samples with a turbidity of greater than 40 NTU must be diluted with turbidity-free distilled water to obtain a reading between 10 and 40 NTU. The turbidity of the original sample is then calculated using the appropriate dilution factor.

15. Metals by Flame Atomic Absorption and ICP:

Metals Group I, Metals Group II, and Barium

a. Samples must be digested as set forth in the approved procedures.

b. Calibrate the instrument each day as directed in the manufacturer's instructions.

c. Each day samples are analyzed, check the instrument calibration by analyzing a blank and a low, medium, and high standard covering the operating range of the instrument.

d. Analyze a quality control standard daily for each parameter.

e. Analyze a duplicate sample daily for each parameter.

16. Metals Hydride:

Arsenic and Selenium

a. Samples must be digested according to the approved procedures.

b. Set up the instrument according to the manufacturer's instructions.

c. Prepare a calibration curve each day by analyzing a blank and a low, medium, and high standard.

d. In addition to the calibration standards, analyze one quality control standard each day samples are analyzed.

e. Analyze one duplicate sample each day samples are analyzed.

17. Arsenic SDDC Colormetric

a. Samples must be digested according to the approved procedures.

b. Prepare a calibration curve by analyzing a blank and a low, medium, and high standard.

c. In addition to the calibration standards analyze one quality control standard each day samples are analyzed.

d. Analyze one duplicate sample daily.

18. Mercury

a. Set up the instrument according to the manufacturer's instructions and the approved procedure.

b. Prepare a calibration curve by analyzing a blank and a low, medium, and high standard each day samples are analyzed.

c. In addition to the calibration standards, analyze a quality control standard daily.

d. Analyze one duplicate sample each day samples are analyzed.

19. Atomic Absorption Furnace

Metals Group I, Metals Group II, Arsenic, Selenium, and Barium

a. Samples must be digested as set forth in the approved procedures.

b. Set up and calibrate the instrument according to the manufacturer's instructions.

c. Each day samples are analyzed, check the instrument calibration by analyzing a blank and a low, medium, and high standard covering the operating range of the instrument.

d. Analyze a quality control standard daily.

e. Analyze a duplicate sample daily.

f. Use of the method of standard additions is recommended for all samples.

20. Analytical Balance

a. The balance must be mounted on a stable surface that will allow accurate weighings of 0.1 mg.

b. Weigh a standard weight each day the balance is used and maintain a log of values read.

c. Check the balance zero before each use.

d. Check the balance with a low, medium, and high standard weight quarterly. Enter results in the balance log.

21. Approved Procedures

a. The EPA approved Federal Register procedures must be used unless a variance has been obtained from EPA.

b. A copy of the approved reference procedures must be available in the laboratory.

22. Chemicals, Reagents, and Glassware

a. Reagents must be prepared and used as detailed in the reference procedures.

b. Date all chemicals received and all reagent solutions prepared.

c. All chemicals should be reagent grade, when available.

d. Maintain a record of all standardizations performed.

e. We recommend that all glassware be Class A, when available.

23. Sample Preservation

a. Samples should be preserved immediately after collection.

b. Document the type of preservatives that are to be used and when samples are preserved.

24. Records

Analytical and quality control records must be available for inspection and include the following:

a. Date samples are collected and date analyzed.

b. Daily lab worksheets and workbooks.

c. Values obtained on standards, blanks, duplicate samples, and standard curves.

d. A record of all required quality controls.

e. All worksheets must contain the signature or initials of the analyst(s) performing that function.

f. All analytical records must be retained for at least three years.

25. Corrective Action

At any time that required quality controls indicate an analytical problem, reflect differences in values greater than allowed by the standard procedures, or differences in values exceed ± 25% of a known value, corrective action must be taken and corresponding samples re-analyzed if possible.

26. Statistical Control Limits

It is recommended that each laboratory calculate statistical control limits, but it is not required at this time.

a. Precisions Control Limits:

Using 30-40 sets of duplicate sample results or an annual data set, calculate precision control limits using the formulas given below:

Range (R) = 1st analysis - 2nd analysis

$$\bar{R} = \frac{\Sigma R}{n}$$

$$UWL_R = 2.51\ \bar{R}$$

$$UCL_R = 3.27\ \bar{R}$$

Where: $\bar{R}$ = average range

$UWL_R$ = Upper Warning Limit

$UCL_R$ = Upper Control Limit

2.51 = Shewhart factor for 2s (duplicate)

3.27 = Shewhart factor for 3s (duplicate)

NOTE: For procedures that have a large concentration range, the duplicate results must be grouped according to the concentration level. For example, BOD samples may be grouped as follows: 0 - 10 mg/1, 10 - 100 mg/1, and greater than 100 mg/1. Precision limits for each range would be calculated.

b. Using 30-40 results from analysis of quality control standards or an annual data set, calculate % recovery, average % recovery, standard deviation and control limits for percent recovery using these formulas:

$$P = \frac{\text{observed}}{\text{known}} \times 100$$

$$\bar{P} = \frac{\Sigma P}{n}$$

$$Sp = \sqrt{\frac{\Sigma P^2 - \frac{(\Sigma P)^2}{n}}{n-1}}$$

$$UCL_p = \bar{P} + 3\,Sp$$
$$UWL_p = \bar{P} + 2\,Sp$$
$$LCL_p = \bar{P} - 3\,Sp$$
$$LWL_p = \bar{P} - 2\,Sp$$

Where:

| | | |
|---|---|---|
| P | = | Percent recovery |
| $\bar{P}$ | = | Average percent recovery |
| n | = | Number of analysis |
| Sp | = | Standard deviation of percent recovery |
| UCLp | = | Upper Control limit for percent recovery |
| UWLp | = | Upper warning limit for percent recovery |
| LCLp | = | Lower control limit for percent recovery |
| LWLp | = | Lower warning limit for percent recovery |

c. Prepare Shewhart control charts for precision and accuracy.

d. Use of Control Limits

(1) These control limits can be used to determine if data is in control on a daily basis. For samples results that fall within these control limits, the established precision or accuracy assessment can be applied to the individual samples of the new sample lot.

(2) For sample results that fall outside the established control limits, the system is out of control, or the established control limits are not applicable to the new data set. Corrective action may require the sample set be analyzed again or that new control limits be established.

(3) If seven successive points fall on the same side of the $\bar{P}$ (center line) of the accuracy control charts, the system is out of control and corrective action must be taken.

e. For further information concerning statistical quality control limits, we recommend securing a copy of EPA Handbook for Analytical Quality Control in Water and Wastewater Laboratories EPA-600/4-79-019. This can be obtained by writing:

Mr. Wade Knight
Quality Assurance Officer
U.S. EPA, Region 4
College Station Rd.
Athens, GA 30613

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

IN REPLY REFER TO
11330
MAIN
4 Jan 85

From: Director, Utilities Branch
To: Director, Natural Resources and Environmental Affairs Division

Subj: NPDES Permit

Encl: (1) NC Dept of NatRes & Comm Development ltr undtd

1. The enclosure is forwarded for your use.

G. S. Johnson, Jr.

G. S. JOHNSON, JR.

NREA

North Carolina Department of Natural Resources & Community Development

James B. Hunt, Jr., Governor

James A. Summers, Secretary

DIVISION OF ENVIRONMENTAL MANAGEMENT

Robert F. Helms
Director

Telephone 919 733-7015

Dear NPDES Permit Administrator:

The Environmental Management Commission approved revised laboratory certification regulations as found in NCAC 2H .0800 following a public hearing, comment response period and changes made in response to public comments. The revised regulations require the certification of all Class III and IV wastewater treatment plant (WWTP) laboratories and Class I and II WWTP laboratories providing analyses for their pretreatment programs.

Attached for your information and use are a copy of the laboratory certification regulations, application form and selected pages of the October 26, 1984 Federal Register containing a listing of approved analytical procedures and other sample information required of NPDES permittees.

All WWTP Laboratories subject to section .0802 of the regulation must submit an acceptable application for certification before June 1, 1985.

Regulation .0804 contains a listing of certifiable parameters. Your facility must be certified for all certifiable parameters, included in the monitoring requirements of your NPDES Permit and/or pretreatment program, if the parameter analysis are performed in your lab. If your State required monitoring analyses are performed by a commercial or other laboratory, please complete only sections I, VII and VIII of the application.

In the October 26, 1984 Federal Register, EPA published a new listing of approved analytical procedures and sample requirements to be used by NPDES permit holders with a January 24, 1985 implementation date. Copies of the Tables listing analytical procedures and sample requirements are provided from the 203 page document. The Table listing supersedes section .0805(a)(1) of the attached regulations and all permittees must conform to the October 26, 1984 requirements by January 24, 1985. We are in the process of changing section .0805(a)(1) of the attached regulation to make it consistent with EPA requirements. A public hearing notice concerning the change will be mailed in the near future. Parameter references in the application are those cited in the October 26, 1984 Federal Register. Note that the new listing includes a procedure for nitrogen inhibited BOD analysis ($CBOD_5$), however, each permittee should continue using the total $BOD_5$ procedure unless otherwise specified on your permit.

Each facility must designate a laboratory supervisor in the application for certification. Part .0805(a)(3) of the regulation sets forth the requirements, however, all existing supervisors will be accepted. The person designated should be the person responsible for the data reported and the solving of any laboratory analytical problems. This could be a laboratory chemist, plant manager, chief operator, plant superintendent, or City manager if their duties so dictate. Following receipt of the application, all correspondence will be directed to the laboratory supervisor.

Prior to certification, each laboratory will be billed the appropriate fees as set forth in section .0806 of the regulations. Certification fees for metals Group I and II will be $20.00 for each group.

The Laboratory Branch is looking forward to working with your laboratory in the certification program. If you have questions or need additional information, contact William B. Edwards, Jr., telephone 919-733-3908.

Sincerely yours,

Robert F. Helms
Director

North Carolina Department of Natural Resources & Community Development
James B. Hunt, Jr., Governor
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Robert F. Helms
Director

NORTH CAROLINA ADMINISTRATIVE CODE

TITLE 15

DEPARTMENT OF NATURAL RESOURCES

AND

COMMUNITY DEVELOPMENT

CHAPTER 2

ENVIRONMENTAL MANAGEMENT DIVISION

SUBCHAPTER 2H

PROCEDURES FOR PERMITS, APPROVALS

SECTION .0800

LABORATORY CERTIFICATION



ENVIRONMENTAL MANAGEMENT COMMISSION
RALEIGH, NORTH CAROLINA

## SECTION .0800 - LABORATORY CERTIFICATION

### .0801 PURPOSE

These Regulations set forth the requirements for state certification of commercial, municipal, and industrial laboratories to perform water analyses, required by the Water and Air Quality Reporting Act, G.S. 143-215.63 et seq; Environmental Management Commission Regulations for Surface Water Monitoring, Reporting, found in Subchapter 2B of this Chapter, Section .0500 and Environmental Management Commission Regulations for Local Pretreatment Programs, found in 15 NCAC 2H .0900.

History Note: Statutory Authority G.S. 143-215.3(a)(1); 143-215.3(a)(10); Eff. February 1, 1976; Amended Eff. December 1, 1984; November 1, 1978.

### .0802 SCOPE

These Regulations apply to commercial laboratories and Class III and IV municipal or industrial wastewater treatment plant laboratories which perform water analyses for persons subject to G.S. 143-215.1, 143-215.63, et seq., or the Environmental Management Commission Regulations for Surface Water Monitoring, Reporting found in Subchapter 2B of this Chapter, Section .0500. These Regulations also apply to all wastewater treatment plant laboratories for municipalities having Local Pretreatment Programs as found in 15 NCAC 2H .0900. Municipal and industrial laboratories that perform analyses for two or less of the parameters listed in Paragraph .0804(a) of these Regulations are exempt from the requirements of these Regulations.

History Note: Statutory Authority G.S. 143-215.3(a)(1); 143-215.3(a)(10); Eff. February 1, 1976; Amended Eff. December 1, 1984.

### .0803 DEFINITIONS

The following terms as used in this Section shall have the assigned meaning:

(1) Commercial Laboratory means any laboratory which is seeking to analyze water samples for others.

(2) State means the North Carolina Division of Environmental Management of the Department of Natural Resources and Community Development, or its successor.

(3) State Laboratory means the laboratory branch of the North Carolina Division of Environmental Management, or its successor.

(4) Unacceptable results on performance evaluation samples or split samples are those that vary by more than plus or minus 25 percent of the value determined by the State Laboratory or the State Laboratory may adopt specific variance limits for a particular parameter.

(5) Certification is a declaration by the state that the personnel, equipment, records, quality control procedures, and methodology cited by the applicant are accurate and that the applicant's proficiency has been considered and found to be acceptable.

(6) Decertification is loss of certification.

(7) Recertification is reaffirmation of certification.

(8) Municipal Laboratory means a laboratory operated by a municipality or other local government to analyze samples from its wastewater treatment plant(s).

(9) Industrial Laboratory means a laboratory operated by an industry to analyze samples from its wastewater treatment plant(s).

(10) Pretreatment Program means a program of waste pretreatment requirements set up in accordance with 15 NCAC 2H .0900 and approved by the Division of Environmental Management.

History Note: Statutory Authority G.S. 143-215.3(a)(1); 143-215.3(a)(10); Eff. February 1, 1976; Amended Eff. December 1, 1984; November 1, 1978.

.0804 PARAMETERS FOR WHICH CERTIFICATION MAY BE REQUESTED

Commercial laboratories need to obtain certification only for parameters which will be reported by the client to comply with the monitoring and pretreatment regulations. Municipal and Industrial Laboratories need to obtain certification only for parameters which will be reported to the state to comply with monitoring and pretreatment regulations. A listing of selected parameters follows:

(1) BOD
(2) COD
(3) Chloride
(4) Coliform, fecal MF
(5) Coliform, total MF
(6) Coliform, fecal tube
(7) Coliform, total tube
(8) Cyanide
(9) Fluoride

(10) Grease and Oil
(11) Hardness, total
(12) MBAS
(13) Metals, Group I
(a) aluminum
(b) beryllium
(c) cadmium
(d) chromium, total
(e) cobalt
(f) copper
(g) iron
(h) lead
(i) maganese
(j) nickel
(k) zinc
(14) Metals, Group II
(a) antimony
(b) silver
(c) thallium
(15) Arsenic
(16) Barium
(17) Mercury
(18) Selenium
(19) Ammonia nitrogen
(20) Total Kjeldahl nitrogen (TKN)
(21) Nitrate plus nitrite nitrogen
(22) Total phosphorus
(23) Orthophosphate
(24) pH
(25) Phenols
(26) Residue, total
(27) Residue, total suspended
(28) Turbidity

History Note: Statutory Authority G.S. 143-215.3(a)(1); 143-215.3(a)(10); Eff. February 1, 1976; Amended Eff. December 1, 1984.

.0805 CERTIFICATION AND RENEWAL OF CERTIFICATION

(a) Prerequisites for Certification

(1) Water. Analytical methods shall conform to those found in 40 CFR-136.2 et seq, Vol. 41 Federal Register, p. 52780 (December 1, 1976) including any amendments through June 1, 1984, as submitted by the Environmental Protection Agency for water programs. 40 CFR-136.2 et seq, Vol. 41 Federal Register lists methods from the

14th Edition of "Standard Methods for the Examination of Water and Wastewater" (published jointly by the American Public Health Association, the American Water Works Association, and the Water Pollution Control Federation) and "Methods for Chemical Analysis of Water and Wastes", 1974 (prepared by the U.S. Environmental Protection Agency). The 15th Edition of "Standard Methods for the Examination of Water and Wastewater" and the 1979 "Methods of Chemical Analysis of Water and Wastes" have reprinted most of the approved procedures. In cases where these references have reprinted the same procedures with no changes, they are acceptable for use in compliance with these Regulations.

(2) Performance Evaluations. Each laboratory must demonstrate satisfactory performance on evaluation samples submitted by the State Laboratory.

(3) Supervisory Requirements.

(A) The supervisor of a commercial laboratory must have a minimum of a B.S. or A.B. degree from an accredited college or university in chemistry or closely related science curriculum plus a minimum of two years laboratory experience in analytical chemistry, or a two year associate degree from an accredited college, university, or technical institute in chemistry technology, environmental sciences, or closely related science curriculum plus a minimum of four years experience in analytical chemistry. Non-degree supervisors must have at least six years laboratory experience.

(B) The supervisor of a municipal or industrial waste water treatment plant laboratory must have a minimum of a B.S. or A.B. degree from an accredited college or university in chemistry or closely related science curriculum plus a minimum of six months laboratory experience in analytical chemistry, or a two year associate degree from an accredited college, university, or technical institute in chemistry technology, environmental sciences, or closely related science curriculum plus a minimum of two years experience in analytical chemistry. Non-degree supervisors must have at least six years laboratory experience.

(C) All laboratory supervisors are subject to review by the State Laboratory. One person may serve as supervisor of no more than two laboratories. The supervisor shall provide personal and direct supervision of the technical personnel and be held

responsible for the proper performance and reporting of all analysis made for these Regulations. If the supervisor is to be absent, the supervisor shall arrange for a substitute capable of insuring the proper performance of all laboratory procedures. Existing laboratory supervisors that do not meet the requirements in this Paragraph may be accepted after review by the State Laboratory and meeting all other certification requirements.

(4) Application. Each laboratory requesting state certification or certification renewal shall submit an application in duplicate to the State Laboratory. Each application will be reviewed to determine the adequacy of personnel, equipment, records, quality control procedures, and methodology. After receiving a completed application and prior to issuing certification, a representative of the State Laboratory may visit each laboratory to verify the information in the application and the adequacy of the laboratory.

(5) Facilities and equipment. Each laboratory requesting certification must contain or be equipped with the following:

(A) A minimum of 150 sq ft of laboratory space;

(B) A minimum of 12 linear feet of laboratory bench space;

(C) A sink with hot and cold water;

(D) Adequate lighting, cooling, and heating;

(E) An analytical balance capable of weighing 0.1 mg, mounted on a heavy shock proof table;

(F) A refrigerator of adequate size that will maintain temperature of 4°C;

(G) An EPA approved or a current copy of "Standard Methods for the Analysis of Water and Wastewater" or EPAs "Methods for Chemical Analysis of Water and Wastes";

(H) A source of distilled or deionized water that will meet the minimum criteria of the approved methodologies;

(I) Glassware, chemicals, supplies, and equipment required to perform all analytical procedures included in their certification.

(6) Analytical Quality Control Program. Each laboratory shall develop and maintain a document outlining the analytical quality control practices used for the parameters included in their certification. Supporting records shall be maintained as evidence that these

practices are being effectively carried out. The quality control program shall be available for inspection by the State Laboratory and include the following:

(A) All analytical quality control data pertinent to each certified analysis must be available for inspection upon request.

(B) Analyze one duplicate sample and one known standard in addition to calibration standards each day samples are analyzed to document precision and accuracy.

(C) Any quality control procedures required by a particular approved method shall be considered as required for certification for that analysis.

(D) All quality control requirements as set forth by the State Laboratory.

(E) A corrective action policy requiring that at any time quality control results indicate an analytical problem, resolve the problem and rerun any samples involved.

(F) A policy requiring that all analytical records must be maintained for a period of three years.

(b) Issuance of Certification

(1) In the absence of substantial deficiencies, certification will be issued by the Director, Division of Environmental Management, Department of Natural Resources and Community Development, or his delegate, for each of the applicable parameters requested.

(2) Initial certifications will be issued for prorated time periods to schedule all certification renewals on the first day of January.

(3) Initial certification shall be valid for up to three years from date of issue.

(c) Maintenance of Certification.

(1) To maintain certification for each parameter, a certified laboratory must analyze up to three performance evaluation samples per year submitted by the State Laboratory as an unknown. Laboratories submitting unacceptable results on a performance evaluation sample may be required to analyze more than three samples per year.

(2) In addition, the State Laboratory may request that samples be split into two equal representative portions, one part going to the state and the other to the certified laboratory for analysis.

(3) A certified laboratory will be subject to periodic inspections during the certification period and shall make time and records available for inspections.

(4) The State Laboratory will maintain a list of certified commercial laboratories and the parameters for which the laboratories have been certified. The list will be revised every six months.

(5) The State Laboratory will maintain a list of certified municipal and industrial laboratories and the parameters for which the laboratories have been certified. The list will be revised every six months.

(6) A certified laboratory must provide the State Laboratory with written notice of laboratory supervisor changes within 30 days of such changes.

(d) Certification Renewals.

(1) Applications for certification renewal will be submitted in duplicate to the State Laboratory 30 days in advance of expiration of certification.

(2) Certification renewals of laboratories shall be issued for three years with the exception that renewals for existing certified laboratories may be prorated to make all certification renewals due on the first day of January.

(e) Discontinuation of Certification

(1) A laboratory may discontinue certification for any or all parameters by making a written request to the State Laboratory.

(2) After discontinuation of certification, a laboratory may be recertified by meeting the requirements for initial certification.

History Note: Statutory Authority G.S. 143-215.3(a)(1); 143-215.3(a)(10); Eff. February 1, 1976; Amended Eff. December 1, 1984; November 1, 1978.

.0826 FEES ASSOCIATED WITH CERTIFICATION PROGRAM

(a) Certification and Certification Renewal Fees. Before being granted certification or certification renewal, laboratories shall pay to the state a fee of twenty dollars ($20.00) for each parameter for which certification is requested, however, the minimum fee will be two hundred fifty dollars ($250.00).

(b) Certification Maintenance Fees. After certification or certification renewal has been issued certified laboratories will pay to the state a certification maintenance fee of two hundred fifty dollars ($250.00) each year. Certification maintenance

fees will not be required for those years in which certification or certification renewal are required. These fees are due on or before the first day of January or the certification anniversary date.

(c) Fees may be prorated in order to make all certification renewals due on the first day of January.

(d) Out-of-state laboratories shall reimburse the state for actual travel and subsistence costs incurred in certification and maintenance of certification.

History Note: Statutory Authority G.S. 143-215.3(a)(1); 143-215.3(a)(10); Eff. February 1, 1976; Amended Eff. December 1, 1984.

.0807 DECERTIFICATION

(a) Laboratory Decertification. Once certified, a laboratory may lose its certification for all parameters by failing to:

(1) Maintain the facilities, or records, or personnel, or equipment, or quality control program as set forth in the application and these Regulations; or

(2) Submit truthful and accurate data reports; or

(3) Pay required fees by the date due; or

(4) Discontinue supplying data for clients or programs described in Rule .0802 of this Section during periods when a parameter decertification is in effect.

(b) Parameter Decertification. Once certified, a laboratory may loose its certification for a a parameter by failing to:

(1) Obtain acceptable results on two consecutive performance evaluation samples submitted by the State Laboratory; or

(2) Obtain acceptable results on three consecutive split samples that have also been analyzed by the State Laboratory; or

(3) Submit a split sample to the State Laboratory as requested ; or

(4) Use approved methods of analysis; or

(5) Report equipment changes that would affect its ability to perform the test within 30 days of such changes; or

(6) Report analysis of performance evaluation samples submitted by the State Laboratory within 30 days of receipt; or

(7) Maintain records and perform quality controls as set forth by these Regulations and the State Laboratory for a particular parameter; or

(8) Maintain equipment required for a particular parameter.

(c) Decertification Requirements.

.0809 RECIPROCITY

(a) Laboratories certified under other state certification programs may be given reciprocity certification where such programs meet the requirements of these Regulations. In requesting reciprocity certification, laboratories shall include with the application required by Regulation .0805(a) of this Section a copy of their certification and Regulation from the certifying agency.

(b) Laboratories certified on the basis of program equivalency shall pay the fees required by Regulation .0806 of this Section.

History Note: Statutory Authority G.S. 143-215.3(a)(1); 143-215.3(a)(10); Eff. February 1, 1976; Amended Eff. December 1, 1984.

.0810 ADMINISTRATION

(a) The Director of the Division of Environmental Management, Department of Natural Resources and Community Development, or his delegate, is authorized to issue certification, to reject applications for certification, to renew certification, to issue recertification, to issue decertification, and to issue reciprocity certification.

(b) Appeals. In any case where the Director of the Division of Environmental Management, Department of Natural Resources and Community Development or his delegate denies certification, or decertifies a laboratory, the laboratory may appeal to the director or his delegate for a hearing. Upon receipt of such a request, the director or his delegate shall convene a hearing of the type provided for Environmental Management Commission Regulation in 15 NCAC 2I .0300, Administrative Hearings. Appeal from the decision of a hearing officer appointed by the director or his delegate shall be governed by the Environmental Management Commission Regulation on administrative hearings.

History Note: Statutory Authority G.S. 143-215.3(a)(1); 143-215.3(a)(10); Eff. February 1, 1976; Amended Eff. December 1, 1984; November 1, 1978.

.0811 IMPLEMENTATION

(a) Commercial Laboratories

(1) Certified commercial laboratories must meet any new requirements set forth herein within 6 months of these Regulations effective date.

(2) Certification fee changes are effective January 1, 1985.

(3) Requests for new parameters can be made by submitting a proper application form.

(b) Municipal and Industrial Laboratories

(1) All Municipal and Industrial Waste Treatment Plant Laboratories subject to Rule .0802 of these Regulations are required to be certified.

(2) All Municipal and Industrial Waste Treatment Plant Laboratories subject to Rule .0802 of these Regulations must submit an application for certification within six months of these Regulations effective date. Laboratories submitting an acceptable application will be considered in compliance with these Regulations until the State Laboratory can process the application and issue or deny certification.

(3) Laboratories that cannot meet initial certification requirements must comply with the Decertification Requirements as set forth in Rule .0807(c) of these Regulations.

History Note: Statutory Authority G.S. 143-215.3(a)(1); 143-215.3(a)(10);
Eff. December 1, 1984.

T-5221/3

State of North Carolina
Department of Natural Resources and Community Development
512 North Salisbury Street • Raleigh, North Carolina 27611

James G. Martin, Governor

S. Thomas Rhodes, Secretary

Division of Environmental Management

APR 5 1985

Dear NPDES Permit Administrator:

RE: Quality Assurance Guidance and Steps Involved in Securing Certification

Enclosed is the quality assurance guidance that was promised in Mr. Helms' January 1985 letter that transmitted wastewater laboratory certification information. Part .0805(a)(6) of the certification regulation states that each laboratory shall develop and maintain a quality assurance document outlining the quality control practices of the laboratory. Laboratories are not expected to have a quality control document, but are expected to begin developing one.

Parts I - III of the enclosed information is general guidance for developing a quality control plan and part IV is a listing of the minimum controls required for certification. Although it will be July or August 1985 before we begin processing certification applications, we encourage each laboratory to begin implementing the required quality controls so they will be better prepared for certification. We will consider substituting existing programs that are not identical to the enclosed required program. For example, laboratories analyzing spiked samples could be exempted from analyzing some of the quality control standards.

Questions have been raised concerning quality controls for parameters for which standards are not readily available (Ex. Coliform). Quality control requirements for these parameters have been modified. At a future public hearing, we will propose changes in the certification regulations that properly address quality controls for these parameters.

Some laboratories have requested information about the steps involved in securing certification. The following is a summary of the steps involved.

1. The laboratory completes the application forms and submits them to the Division of Environmental Management (DEM) Laboratory.

2. DEM Laboratory reviews the applications, notes any deficiencies and returns the application for any needed information.

P.O. Box 27687, Raleigh, North Carolina 27611-7687 Telephone 919-733-4984

3. DEM Laboratory mails performance evaluation samples to the laboratories seeking certification. This will begin in July or August 1985.

4. Your laboratory analyzes the samples and submits results to the DEM Laboratory.

5. DEM Laboratory reviews and evaluates these results and, if necessary, sends rerun samples. The DEM Laboratory will assist any laboratories having problems producing acceptable results. Assistance may be in the form of known samples, methodology, work forms, or quality controls.

6. The DEM Laboratory schedules and performs an onsite laboratory inspection. Time sensitive samples may be delivered during the inspection.

7. An inspection report listing any observed deficiencies will be prepared and mailed.

8. The laboratory seeking certification agrees to take any required corrective actions. Reasonable time will be given for taking corrective actions.

9. The laboratory requesting certification is billed the appropriate fees.

10. Upon receipt of payment, DEM issues certification for up to three years.

We continue to look forward to working with your laboratory in the certification program. If you have any questions concerning the laboratory certification program, contact Mr. Billy D. Byrd or Mr. William B. Edwards, Jr. at 919-733-3908.

Sincerely,

W. B. Edwards, Jr.

William B. Edwards, Jr.

Enclosure

March 1985

# NRCD/DEM Wastewater Laboratory Certification
# Guidance for Preparing a Quality Assurance Document

## I. Introduction

All certified laboratories must be committed to producing quality assured data and carrying out the necessary quality controls to qualify data produced. It must be recognized that the additional controls will result in an increase in operating cost and will require additional work time. The guidance outlined here is based on the analysis of known standards to document accuracy and duplicate samples to document precision. This program also includes documentation of other standard operating procedures.

## II. Sample Receiving and Sample Identification

Each laboratory must have some system of sample identification that will keep each sample discrete. This may be an elaborate sample logging and numbering system for the larger laboratories or simply labeling the samples as influent, effluent, etc., for the smaller laboratories. Also included in this section should be instructions as to what will be done with the samples upon receipt in the laboratory. For example, samples may be preserved and stored for future analysis or they may be taken directly to the laboratory bench and analyzed.

## III. General Laboratory Practices

In order to produce quality data, the analyst must have adequate facilities, services, instrumentation, and supplies and the analyst must properly use and maintain each of these. This section should include general instructions for operating, maintaining and cleaning laboratory apparatus and equipment, and storage of chemicals.

## IV. Quality Controls

Listed below are the minimum quality controls required for North Carolina Wastewater Certification. Some laboratories are already exceeding the controls listed here and are encouraged to continue at that level. We will consider substituting existing programs that are not identical to the items listed here. For example, laboratories analyzing spiked samples could be exempted from analyzing some of the quality control standards.

### 1. BOD

a. The temperature of the BOD incubator must be maintained at 20 ± 1°C using an accurate thermometer inserted in a BOD bottle inside the incubator.

b. Check and record the incubator temperature each day.

c. Calibrate the dissolved oxygen meter each day before analyzing samples and check calibration after completing each group of analyses. (USE Blank)

d. Samples which have a low or high pH, contain chlorine, or other toxics, must be pretreated as described in the approved procedure. After pretreatment, the samples must be seeded to provide an adequate biological population capable of oxidizing the organic material in the sample.

e. Perform a glucose-glutamic acid check each day seeded samples are analyzed.

f. Sufficient seed must be used to yield a seed correction of 0.6-1.0 mg/l.

g. Each day determine the BOD of the seed material the same as for any other unseeded sample. Calculate the seed correction from the results of the seed BOD. Do not use a seeded blank as the seed correction.

h. Analyze samples using a dilution series that will yield a dissolved oxygen usage of at least 2 mg/l or a residual of at least 1 mg/l.

i. Analyze a duplicate sample daily.

j. Perform a blank dilution water control analysis along with each batch of samples analyzed.

2. COD - Titration Procedure

a. Standardize the COD titrant each day samples are analyzed.

b. Analyze a distilled water blank with each group of samples and make proper corrections.

c. Analyze a quality control standard along with each group of samples analyzed.

Note: A 250 mg/l COD standard may be made by dissolving 0.2125 g potassium acid phthalate (that has been dried at 120°C) in one liter of distilled water. A 25 mg/l COD standard for the low level procedure may be prepared by diluting 10.0 ml of the above solution to 100 mls.

d. Analyze a duplicate sample daily.

e. Use the low level procedure for the analysis of samples with a COD of <50 mg/l.

3. COD - Colormetric

a. Prepare a standard curve as set forth in the standard procedure. As a minimum, the curve must consist of a blank and three standards (low, medium, and high).

b. In addition to the calibration standards, analyze a quality control standard each day.

c. Analyze a duplicate sample daily.

4. Coliform

a. Check the temperature of all incubators daily and maintain a log of values read.

b. The 44.5°C waterbath must be equipped with a thermometer graduated in 0.1°C increments.

c. The 35°C incubator must be equipped with a thermometer graduated in at least 0.5°C increments.

d. Log the maximum temperature and pressure of the autoclave once during each use.

e. Analyze a dilution water blank at the beginning and end of each group of samples analyzed.

f. Analyze one duplicate sample each day.

5. Chloride

a. Standardize the titrant each day samples are analyzed by titrating a sodium chloride standard.

b. Analyze a distilled water blank each day and make proper corrections.

c. Analyze one duplicate sample each day.

6. Hardness

a. Standardize the titrant each day samples are analyzed by titrating a calcium carbonate standard.

b. Analyze a distilled water blank each day samples are analyzed.

c. Analyze one duplicate sample daily.

7. Colormetric Analyses:

Cyanide, Phenol, Colormetric Fluoride, MBAS, Colormetric TKN, Colormetric Ammonia, Nitrate + Nitrite Nitrogen, Total Phosphorus, and Orthophosphate.

a. Each analyst performing the analysis must produce a standard curve consisting of at least a blank and three standards (low, medium, and high).

b. Analyze a blank and a mid-range standard along with each group of samples analyzed. If there is a significant difference in the standard analyzed and the standard curve, resolve the discrepancies or produce a new standard curve.

c. Analyze a duplicate sample with each group of samples analyzed.

8. Ammonia and Total Kjeldahl Nitrogen - Titration Procedure

a. Analyze a distilled water blank each day samples are analyzed.

b. For ammonia, analyze one ammonium chloride standard each day samples are analyzed.

c. For TKN, analyze one organic nitrogen standard each day samples are analyzed.

Note: A 100 mg/l organic nitrogen stock standard can be prepared by dissolving 1.0503 g of glutamic acid in 600 ml distilled water containing 1 ml concentrated $H_2SO_4$ and diluting to one liter. Diluting 10 ml of this standard and 1 ml concentrated $H_2SO_4$ to one liter with distilled water will yield a solution containing 1 mg/l of nitrogen.

d. Analyze a duplicate sample daily.

9. Electrode Procedure:

Fluoride, Ammonia Nitrogen, and Total Kjeldahl Nitrogen

a. Calibrate the meter according to the manufacturer's instructions.

b. Check the meter calibration by analyzing a medium level quality control standard each day.

c. Analyze a duplicate sample each day samples are analyzed.

10. Automated Procedures:

Ammonia Nitrogen, Total Kjeldahl Nitrogen, Nitrate + Nitrite Nitrogen, Total Phosphorus, and Orthophosphate

a. Calibrate the instrument according to the manufacturer's instructions.

b. Check the instrument calibration each day by analyzing a low, medium, and high standard.

c. Analyze a quality control standard after every ten samples and at the end of each group of analyses.

d. Analyze one duplicate sample each day samples are analyzed.

e. For TKN, analyze one organic nitrogen quality control standard each day samples are analyzed.

11. Oil & Grease

a. Perform a blank analysis on each batch of freon used and make proper corrections.

b. The freon must be distilled from the extraction flask using a water bath controlled at 70°C.

c. The extract must be filtered through Whatman #40 filter paper or equivalent.

d. It is recommended that a reference standard be analyzed quarterly.

12. pH

a. Standardize the meter using a low and high buffer daily or before each use.

b. It is recommended that a reference standard be analyzed quarterly.

c. Analyze a duplicate sample daily.

13. Total Residue and Total Suspended Residue

a. Check and record drying oven temperature each day used.

b. Analyze one duplicate sample each day samples are analyzed.

c. It is recommended that blank dishes and crucibles be carried through the entire procedure to determine if proper cooling times are being used.

d. It is recommended that a reference standard be analyzed quarterly.

14. Turbidity

a. Standards as described in the approved procedure must be secured and used.

b. Each day the turbidimeter is used, calibrate it with at least one standard for each instrument range used.

c. Analyze one duplicate sample each day samples are analyzed.

d. Samples with a turbidity of greater than 40 NTU must be diluted with turbidity-free distilled water to obtain a reading between 10 and 40 NTU. The turbidity of the original sample is then calculated using the appropriate dilution factor.

15. Metals by Flame Atomic Absorption and ICP:

Metals Group I, Metals Group II, and Barium

a. Samples must be digested as set forth in the approved procedures.

b. Calibrate the instrument each day as directed in the manufacturer's instructions.

c. Each day samples are analyzed, check the instrument calibration by analyzing a blank and a low, medium, and high standard covering the operating range of the instrument.

d. Analyze a quality control standard daily for each parameter.

e. Analyze a duplicate sample daily for each parameter.

16. Metals Hydride:

Arsenic and Selenium

a. Samples must be digested according to the approved procedures.

b. Set up the instrument according to the manufacturer's instructions.

c. Prepare a calibration curve each day by analyzing a blank and a low, medium, and high standard.

d. In addition to the calibration standards, analyze one quality control standard each day samples are analyzed.

e. Analyze one duplicate sample each day samples are analyzed.

17. Arsenic SDDC Colormetric

a. Samples must be digested according to the approved procedures.

b. Prepare a calibration curve by analyzing a blank and a low, medium, and high standard.

c. In addition to the calibration standards analyze one quality control standard each day samples are analyzed.

d. Analyze one duplicate sample daily.

18. Mercury

a. Set up the instrument according to the manufacturer's instructions and the approved procedure.

b. Prepare a calibration curve by analyzing a blank and a low, medium, and high standard each day samples are analyzed.

c. In addition to the calibration standards, analyze a quality control standard daily.

d. Analyze one duplicate sample each day samples are analyzed.

19. Atomic Absorption Furnace

Metals Group I, Metals Group II, Arsenic, Selenium, and Barium

a. Samples must be digested as set forth in the approved procedures.

b. Set up and calibrate the instrument according to the manufacturer's instructions.

c. Each day samples are analyzed, check the instrument calibration by analyzing a blank and a low, medium, and high standard covering the operating range of the instrument.

d. Analyze a quality control standard daily.

e. Analyze a duplicate sample daily.

f. Use of the method of standard additions is recommended for all samples.

20. Analytical Balance

a. The balance must be mounted on a stable surface that will allow accurate weighings of 0.1 mg.

b. Weigh a standard weight each day the balance is used and maintain a log of values read.

c. Check the balance zero before each use.

d. Check the balance with a low, medium, and high standard weight quarterly. Enter results in the balance log.

21. Approved Procedures

a. The EPA approved Federal Register procedures must be used unless a variance has been obtained from EPA.

b. A copy of the approved reference procedures must be available in the laboratory.

22. Chemicals, Reagents, and Glassware

a. Reagents must be prepared and used as detailed in the reference procedures.

b. Date all chemicals received and all reagent solutions prepared.

c. All chemicals should be reagent grade, when available.

d. Maintain a record of all standardizations performed.

e. We recommend that all glassware be Class A, when available.

23. Sample Preservation

a. Samples should be preserved immediately after collection.

b. Document the type of preservatives that are to be used and when samples are preserved.

24. Records

Analytical and quality control records must be available for inspection and include the following:

a. Date samples are collected and date analyzed.

b. Daily lab worksheets and workbooks.

c. Values obtained on standards, blanks, duplicate samples, and standard curves.

d. A record of all required quality controls.

e. All worksheets must contain the signature or initials of the analyst(s) performing that function.

f. All analytical records must be retained for at least three years.

25. Corrective Action

At any time that required quality controls indicate an analytical problem, reflect differences in values greater than allowed by the standard procedures, or differences in values exceed ± 25% of a known value, corrective action must be taken and corresponding samples re-analyzed if possible.

26. Statistical Control Limits

It is recommended that each laboratory calculate statistical control limits, but it is not required at this time.

a. Precisions Control Limits:

Using 30–40 sets of duplicate sample results or an annual data set, calculate precision control limits using the formulas given below:

Range (R) = 1st analysis - 2nd analysis

$$\bar{R} = \frac{\Sigma R}{n}$$

$$UWL_R = 2.51\ \bar{R}$$

$$UCL_R = 3.27\ \bar{R}$$

Where: $\bar{R}$ = average range

$UWL_R$ = Upper Warning Limit

$UCL_R$ = Upper Control Limit

2.51 = Shewhart factor for 2s (duplicate)

3.27 = Shewhart factor for 3s (duplicate)

NOTE: For procedures that have a large concentration range, the duplicate results must be grouped according to the concentration level. For example, BOD samples may be grouped as follows: 0 - 10 mg/1, 10 - 100 mg/1, and greater than 100 mg/1. Precision limits for each range would be calculated.

b. Using 30-40 results from analysis of quality control standards or an annual data set, calculate % recovery, average % recovery, standard deviation and control limits for percent recovery using these formulas:

$$P = \frac{\text{observed}}{\text{known}} \times 100$$

$$\bar{P} = \frac{\Sigma P}{n}$$

$$Sp = \sqrt{\frac{\Sigma P^2 - \frac{(\Sigma P)^2}{n}}{n-1}}$$

$$UCL_p = \bar{P} + 3\,Sp$$

$$UWL_p = \bar{P} + 2\,Sp$$

$$LCLp = \bar{P} - 3\,Sp$$

$$LWLp = \bar{P} - 2\,Sp$$

Where:

| | | |
|---|---|---|
| P | = | Percent recovery |
| $\bar{P}$ | = | Average percent recovery |
| n | = | Number of analysis |
| Sp | = | Standard deviation of percent recovery |
| UCLp | = | Upper Control limit for percent recovery |
| UWLp | = | Upper warning limit for percent recovery |
| LCLp | = | Lower control limit for percent recovery |
| LWLp | = | Lower warning limit for percent recovery |

c. Prepare Shewhart control charts for precision and accuracy.

d. Use of Control Limits

(1) These control limits can be used to determine if data is in control on a daily basis. For samples results that fall within these control limits, the established precision or accuracy assessment can be applied to the individual samples of the new sample lot.

(2) For sample results that fall outside the established control limits, the system is out of control, or the established control limits are not applicable to the new data set. Corrective action may require the sample set be analyzed again or that new control limits be established.

(3) If seven successive points fall on the same side of the $\bar{P}$ (center line) of the accuracy control charts, the system is out of control and corrective action must be taken.

e. For further information concerning statistical quality control limits, we recommend securing a copy of EPA Handbook for Analytical Quality Control in Water and Wastewater Laboratories EPA-600/4-79-019. This can be obtained by writing:

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